

ASPHALTS AND ALLIED SUBSTANCES

*Their Occurrence, Modes of Production,
Uses in the Arts and Methods of Testing*

By

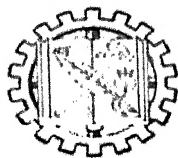
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VOLUME TWO

METHODS OF TESTING;
BIBLIOGRAPHY AND REFERENCES



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PART VI
METHODS OF TESTING

CHAPTER XXXI

SAMPLING

The methods which follow are suitable for sampling the following classes of bituminous materials:

- (A) Crude, Refined and Blended Bituminous Substances.
- (B) Bituminous Paving Materials.
- (C) Bituminized Paper and Fabrics.
- (D) Bituminous Lacquers, Cements, Varnishes and Japans; also Bituminous Emulsions.

For sampling purposes, bituminous materials may be classified according to their physical condition, as follows:

- (1) Liquids to semi-liquids.
- (2) Highly viscous substances.
- (3) Coherent semi-solids to solids.
- (4) Lumpy solids in crushed fragments or powdered form.
- (5) Sheet or roll form.

These methods apply to material in the following types of containers:

- (a) Cans or small containers (up to 10 U. S. gal., inclusive);
- (b) Drums or barrels; (c) Tank cars or tank trucks; (d) Shore tanks; (e) Ship tanks or barge tanks; (f) Pipe lines; (g) Bags;
- (h) Cakes; (i) Bulk or loose form.

Samples are taken for the following types of tests:

- (a) General quality tests; (b) Definite chemical and physical tests; (c) Special quality tests.

Samples are used for one or more of the following purposes:

(a) Visual examination; (b) Laboratory test; (c) Preservation for record; (d) Check and referee tests; (e) The extent of variation of the quality in different portions of the lot; (f) The average quality of the whole lot of material.

Samples should be marked, recorded, and handled throughout in accordance with all the usual precautions of the best laboratory technique.

DEFINITIONS¹

*Average Sample.** An average sample would be one so taken as to contain parts from all sections of the container or pipe in proportion to the volume of each part.

All-levels Sample.† An all-levels sample is one obtained by submerging a closed sample container to a point as near as possible to the draw-off level, opening it and raising it at such a rate that it will be nearly but not quite full when withdrawn.

Upper Sample. An upper sample is one taken at a point 10 per cent of the depth of a uniform cross-section vessel or 10 per cent of the diameter of the horizontal cylindrical tank below the surface.

Middle Sample. A middle sample is one taken at half the depth of the material.

Lower Sample. A lower sample is one taken at a point 10 per cent of the depth of a uniform cross-section vessel or at 10 per cent of the diameter of a horizontal cylindrical tank above the bottom of the vessel.

Composite Sample. A composite sample is a mixture of upper, middle and lower samples containing, for vessels of different shapes,

* It is practically impossible to obtain an average sample, except, perhaps, through a continuous sampling connection from a vertical run in a pipe line with specially constructed draw-off pipes, or by vigorously agitating and stirring the contents of a vessel and drawing off a sample while the contents are still agitated. Samples obtained by lowering and raising a bottle while allowing it to fill have been considered average samples, but obviously the proportions are not related to the tank volumes at the various levels.

† The all-levels sampling method is widely used for ship tanks, barges and large shore tanks. The method can not, however, give an average or a strictly representative sample, not only because the tank volume may not be proportional to the depth and because the operator can not move the sample container with a uniform speed, but also because the rate of filling the sample container is proportionate to the square root of the depth of immersion of the sample container opening below the surface.

volume proportions which correspond approximately to the volumes of the material at these levels, as follows:

	Composite Sample Proportions	
	Uniform Cross Section Vessels	Horizontal Cylindrical Tanks (Assumed Full)
Upper sample.....	1 part	1 part
Middle sample.....	3 parts	8 parts
Lower sample.....	1 part	1 part

For horizontal cylindrical tanks that are only partially filled, the simple geometrically proportioned sampling levels, and composite sample mixtures stated above cannot give samples that are substantially proportionate to the volumes at the different depths, unless the tanks are filled to at least 80 per cent of the diameter.

In referee tests, a mutually satisfactory selection of sampling levels and sample quantities at each level shall be mutually agreed upon, if any departure from the methods specified is to be allowed.

Table CIII gives a set of substantially correct sampling levels and sample quantities for partially filled horizontal cylindrical tanks, which may be used in the absence of other arrangements, em-

TABLE CIII

SAMPLING LEVELS AND SAMPLE QUANTITIES FOR PARTIALLY FILLED HORIZONTAL CYLINDRICAL TANKS

Liquid Depth, Percentage of Diameter	Sampling Level, Percentage of Diameter above Bottom			Quantity of Sample to be Taken at Each Sampling Level		
	Upper	Middle	Lower	Upper	Middle	Lower
1.....			8			10 parts
10.....			10			10 parts
30.....		20	10		6 parts	4 parts
40.....		25	10		7 parts	3 parts
50.....		30	10		8 parts	3 parts
60.....	55	35	10	1 part	8 parts	1 part
70.....	65	40	10	1 part	8 parts	1 part
80.....	75	45	10	1 part	8 parts	1 part
90.....	85	50	10	1 part	8 parts	1 part
100.....	90	50	10	1 part	8 parts	1 part

ploying values in the tabulation that lie nearest to the *actual filling depth*.

Continuous Sample. A continuous sample is one obtained from a pipe or conduit conveying the material, in such a manner as to give at all times a representative average of all the sections and section velocities of the stream throughout the period of transit.

Dipper Sample. A dipper sample is one taken by interposing a dipper or collecting vessel into the path of a free flowing stream, so as to collect a definite volume from the full cross-section of the stream at regular time intervals, or at time intervals so varied as to obtain samples in proportion to the amounts being delivered.

Mixed Sample. A mixed sample is one obtained by mixing or vigorously stirring the contents of the original contained and pouring or drawing off the quantity desired.

Thief Sample. A thief sample is one taken by means of a sampling tube from a specified point in the containing vessel being sampled.

*Drain Sample.** A drain sample is one taken by opening the draw-off or the discharge valve and drawing off a suitable portion of the material.

*Bottom Sample.** A bottom sample is one obtained by collecting a portion of the material lying on the bottom surface of the tank, container or line at its lowest point.

Borings Sample. A borings sample is one obtained by collecting the chips made by boring holes with a ship-auger from top to bottom of the material contained in a barrel, case, bag, or cask.

Grab Sample. A grab sample is one obtained by collecting representative portions from loose solids in equal quantities from each part or package of a shipment, in sufficient amount to be representative of all the sizes and components.

GENERAL DIRECTIONS AND PRECAUTIONS

Official samples shall be taken by, or under the immediate supervision of, a person of judgment, skill and previous experience in sampling. The portions taken for samples shall represent the

*Occasionally, as in tank cars, the drain sample will also give a bottom sample. Drain samples and bottom samples are usually taken only in order to examine for moisture, sludge, scale, etc., and in some cases for the purpose of drawing off any free water or extraneous material.

general character and average condition of the lot sampled. If there is any substantial reason indicating that a sample or set of samples may not be fully representative, another sample or set of samples shall be taken.

Great care and good judgment are necessary in taking and handling samples from crude or unrefined materials, not only because of possible segregation, sludging and volatilization, but because of the bearing upon value, refining problems, and methods of handling. A high degree of care shall be employed in handling samples from semi-refined materials upon which definite chemical and physical tests are usually made. The utmost precautions and the most meticulous care shall be exercised in handling samples from refined products, upon which special quality tests are usually made.

For all grades of materials, precautions shall be taken to assure that the sampling apparatus and the samples themselves are neither contaminated with *nor altered by any material* not representative of the lot being sampled.

The operator engaged in sampling shall have clean hands, free from any material (unless it be the material being sampled). Cleaned gloves may be worn, but only when absolutely necessary, as in extremely cold weather, or in handling materials at high temperatures, or materials involving some health or other hazard.

The sampling apparatus shall be thoroughly clean and dry, and free from any substance that will dissolve into and/or color water-white gasoline.

NOTE.—Exception: Crude and dark-colored semirefined materials (such as dark-colored fuel oils) when fluid enough to drain completely from sampling apparatus (Saybolt universal viscosity less than 250 at 100° F.) may be sampled repeatedly with apparatus that has been hung up and thoroughly drained.

Samples shall be permanently marked (preferably with a pencil hard enough to dent the paper)* immediately after collection, with the date, also the hour and minute for continuous and dipper samples, name of the sampler, name or number and owner of vessel, barge, car or container; material sampled, and a reference symbol or number. Thin aluminum strips stamped with the sample number and other information are acceptable. The reference symbol or

* Soft pencil and ink markings are liable to obliteration from moisture, handling or smearing with oil.

number and other markings should be entered in a bound record book.

Sample containers shall be closed immediately after collection. Sample containers shall be of such types as will protect the sample from contamination or deterioration. Specific precautions shall be taken as follows:

(a) Corks, for bottles, shall be clean, free from holes and loose bits of cork.

(b) Sealing wax, or paraffin, shall *not* be poured over corks after filling.

(c) Light-sensitive samples, when placed in bottles, shall be wrapped or otherwise covered at once, to protect them from light.

(d) Tin cans with screwed or overlapped and soldered caps are acceptable, but only if the inside has been scrupulously cleaned and known to be free from dirt, water, washing compounds, naphtha or other solvents, soldering flux or acids, corrosion, rust, and pin holes.

(e) Refined materials should be further protected by covers of paper or metal-foil over the stopper and top to keep moisture and dust away from the filling and emptying opening, and to protect it from contact with the hands. Covers also prevent sealing substances, such as wax or paraffin, from getting on the lip when the seal is made or later opened, and so getting into the sample when it is poured out.

(f) Samples shipped by mail, express or messenger shall be well packed to avoid spillage, leakage, breakage, and loss by evaporation or alteration while in transit.

Volatile samples shall be protected from evaporation while being collected, particularly in the case of continuous and dipper samples, and at once placed in closed containers. Samples shall, if possible, be taken at the time of loading of a shipment and at the point of origination of a shipment. When sampling at the point of origin has not been provided for, or is not feasible, then the sampling shall be carried out as soon as possible after receipt of the shipment.

Number of Packages Sampled. Shipments consisting of a number of separate packages, such as cans, drums, barrels, or boxes, shall have a number opened and sampled equal to the cube root (or the next larger whole number) of the total number of packages in the lot.

Quantity and Subdivision of Samples. (a) *Individual Liquid Samples.* Individual liquid samples taken with a beaker, bottle, or dipper shall be approximately 1 qt. or 1 liter.

(b) *Composite Liquid Samples.* Composite liquid samples taken from vessels of all kinds and sizes shall be at least 5 qt. (or 5 liters) for vessels of uniform cross-section and 10 qt. (or 10 liters) for horizontal cylindrical tanks.

(c) *Gross Liquid Mixed Cargo Samples.* Gross liquid mixed cargo samples taken from the various ship tanks shall have a total gross quantity corresponding to the number of tanks, that is, multiples of 5 to 10 qt., depending on the tank shapes.

(d) *Liquid Samples Taken by the Continuous Method or by the Dipper Method.* Liquid samples taken by the Continuous Method, or by the Dipper Method, shall be approximately 0.1 per cent of the total quantity shipped, but not less than 5 U. S. gal. (or 20 liters) nor more than 40 U. S. gal. (or 160 liters).

(e) *Soft Solid Samples (Waxes, etc.).* Soft solid samples taken by the Borings Method shall consist of three sets of borings $\frac{3}{4}$ in. in diameter and approximately the depth of the material.

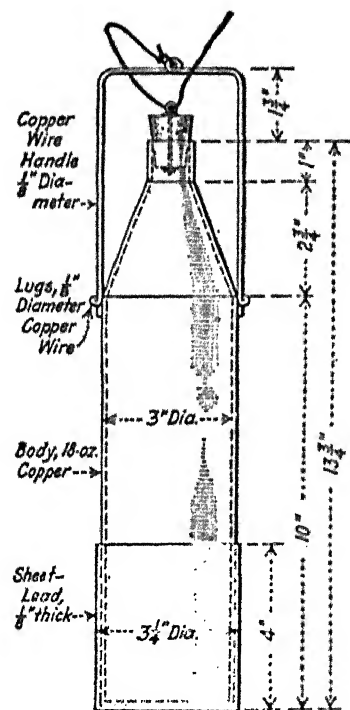
(f) *Lumpy Solid Samples.* Lumpy solid samples taken by the Grab Method shall be equivalent to approximately 0.1 per cent of the lot, but not less than about 50 lb. (25 kg.) nor more than 1000 lb. (500 kg.).

(g) *Subdivision of Gross Samples.* Subdivision of gross samples shall be carried out in the laboratory and should be performed by someone from the laboratory staff, rather than by the person who originally took the samples. The subdivision shall be carried out in accordance with careful laboratory practice and the detailed directions given as a part of the procedure for each method.

(A) SAMPLING CRUDE, REFINED AND BLENDED BITUMINOUS SUBSTANCES

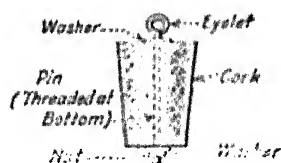
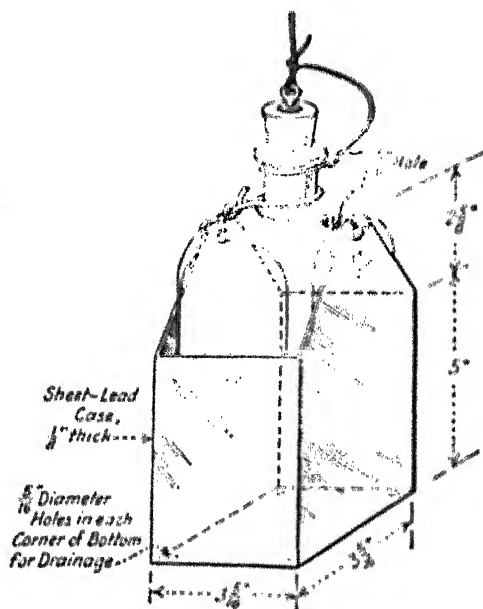
The following procedure² is recommended for sampling all types of crude, refined and blended bituminous substances in various physical conditions.

Care shall be taken that the samples are not contaminated with dirt or any other extraneous matter and that the sampler containers



Weighted Beaker.

Note: This Container Should be Used when one Composite Sample is desired for each Grade of Cargo.

Detail of Cork
Arrangement.

Weighted Bottle.

Note: This Bottle to be used for Obtaining the Individual Samples when the Samples are to be kept dry state, that is, where the Sample must be delivered in the dry form in the same condition as it is contained.

Courtesy A. S. T. M.

FIG. 211.—Standard Sampling Containers for Bottle or Beaker Sampling.

are perfectly clean and dry before filling. Immediately after filling, the sampler containers shall be tightly closed and properly marked for identification on the container itself or on a linen tag attached to the container.

(a) Whenever practicable, bituminous materials shall be sampled at the point of manufacture, and at such time as to allow the tests, controlling acceptance or rejection, to be made in advance of shipment.

(b) When impracticable to take samples at the point of manufacture, they should be taken from the shipment immediately upon delivery.

For routine laboratory examination of acceptability of a given lot, not less than one quart of material should be submitted, which should be representative of the average sample collected as herein-after described. Containers for liquid bituminous materials shall be small-mouth cans with cork-lined screw caps. Containers for semi-solid and solid materials shall be friction-top cans.*

Sampling at Place of Manufacture

The inlet and outlet to the storage tank shall be sealed and a 1-gal. sample drawn from the top, middle and bottom contents. The sample may be taken from drain cocks on the side of the tank, if such are available. Enough material should be discarded to insure a representative sample. Otherwise, samples may be taken by lowering weighted bottles or beakers into the material.⁸ The sampling bottle and sampling beaker shall be made substantially in accordance with the form and dimensions shown in Fig. 211, and the bottle or can should be fitted with a stopper which can be removed by a string or wire attached to it after it has been lowered to the proper depth. The three samples from bulk storage shall be tested separately for consistency in order to detect stratification. They may then be combined and thoroughly mixed for other tests that may be required. A bottle sampler for tank cars is illustrated in Fig. 212. The following instructions shall apply to a substantially full tank:

* Semi-solid to solid bituminous materials when sampled may be conveniently transported to the laboratory in tin cans or boxes, the inside of which has been amalgamated with mercury, to which they do not adhere.

(a) An upper sample shall be taken by lowering the weighted stoppered bottle or beaker till its mouth is 10 per cent of the depth of a tank of uniform cross-section or diameter of a horizontal cylindrical tank below the surface, uncorking it by a quick jerk, allowing it to fill completely, as evidenced by cessation of air bubbles, and then immediately withdrawing it.

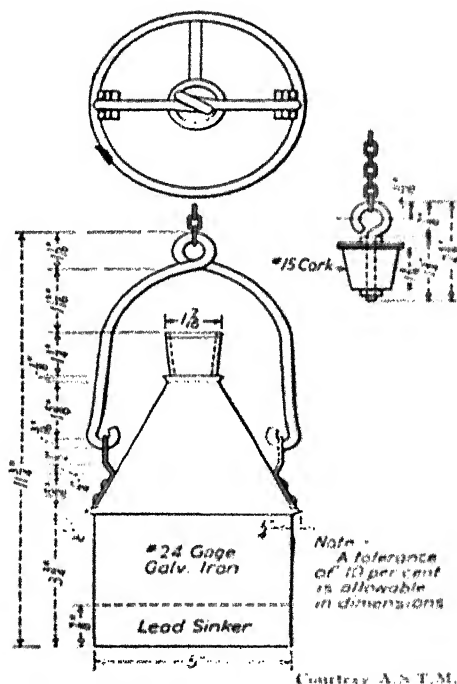


FIG. 212.—Bottle Sampler for Tank Cars.

(b) Three middle samples shall be taken in a similar manner from a tank of uniform cross-section and eight middle samples from a horizontal cylindrical tank with the bottle or beaker mouth lowered to the middle of the tank.

(c) A lower sample shall be taken in a similar manner with the bottle or beaker mouth lowered to a point 10 per cent of the depth of a tank of uniform cross-section, or diameter of a horizontal cylindrical tank above the bottom of the tank.

(d) The upper, middle, and lower samples from each tank car,

tank truck, shore tank, individual ship tank or barge tank shall be combined into a composite sample.

(e) The composite samples taken with the beaker from the various ship tanks shall be combined into a mixed cargo sample.

(f) The beaker samples shall be poured as taken into a clean 5 or 10-gal. can, or cans, and the cans closed and covered, labeled and delivered to the laboratory, shaken or mixed with the churn-dasher type of stirrer, and portions withdrawn by means of a thief for test.

(g) The composite samples taken with the bottle from the various ship tanks shall be kept separate and delivered separately to the laboratory.

(h) The bottle samples shall all be delivered to the laboratory in the bottle in which they were taken.

(i) When loading or discharging any finished product, samples shall also be taken from the shore tanks and at frequent intervals from the shore pipe line on the dock. These samples shall also be tested whenever it seems advisable.

(j) A portion of all samples from all shipments shall be fully labeled and kept in storage until the final disposition of the material.

(k) Ship and barge tanks should be sampled not only immediately after loading, but also before discharging.

Where tank cars, distributors or barrels are being filled, samples may conveniently be taken from the pipe line through which the material is flowing, as hereinafter described.

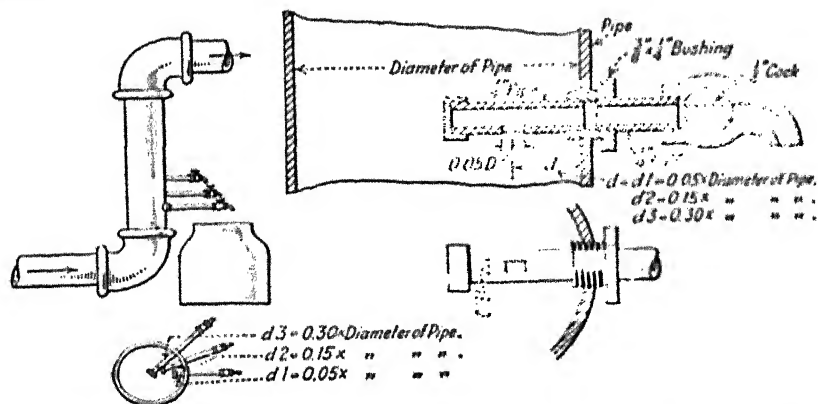
When Material Is Pumped under Pressure. In the case of pipe lines, filling lines and transfer lines, the continuous method of sampling⁴ is applicable. The continuous sampling connection shall be constructed and installed in accordance with the form and dimensions shown in Fig. 213, and the churn-dasher form a stirrer as shown in Fig. 214. The three plug-cocks shall be opened, as nearly as possible, to the same angle of opening, so that a steady stream is drawn off from each cock and at such a rate as will allow about 0.1 per cent of the stream to be diverted during the passage of the lot being sampled, but not more than 40 U. S. gal. The following precautions should be observed:

(a) For semi-liquid materials, the receiver, or receivers, shall be kept warm by means of a steam-coil adjusted to keep the material just above the liquefying temperature.

(b) The receiver, or receivers, should be kept covered and

closed, except for their vents and filling stream openings, or pipes.

(c) Screens of larger area than the openings may be placed around the $\frac{1}{4}$ -in. pipes, or $\frac{1}{4}$ -in. screen fittings inserted in the drip lines.



Courtesy A. S. T. M.

FIG. 213.—Continuous Sampling Connection.

(d) A pipe-cap with a suitable orifice hole may be substituted for the cocks, where the orifice size has been predetermined so as to give the right amount of sample. The sampling pipe shall be inserted into a rising section of the pipe line on the discharge side of

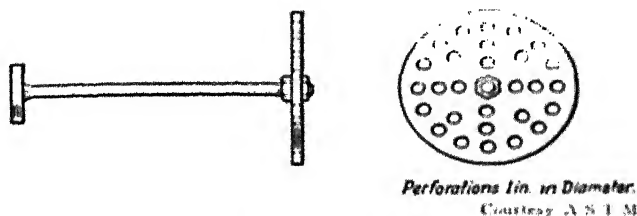


FIG. 214.—Churn-Dasher Type of Stirrer.

the pump. The sampling pipe shall be not more than one-eighth the diameter of the line pipe and its opening should be turned at an angle of 90 deg. facing the flow of the liquid. This pipe shall be provided with a plug cock and shall discharge into a receiving drum of 50-gal. capacity. The plug cock shall be so adjusted that there is a steady continuous flow of bituminous material through it

and shall be so regulated that the receiver will fill in the same time that is required to make the entire pumping. In the case of semi-solid materials, the receiver shall be provided with a steam coil which shall keep the contents at a temperature just above the liquefying point. At completion of the pumping, the receiver shall be thoroughly agitated, and a 1-qt. sample taken therefrom. The sampling shall be so regulated that for each 1000 gal. of material pumped, at least 1 gal. of sample is taken; but 40 gal. shall be the limit required for any one cargo. Care should be taken that the drip-cock, or pipe-cap, after once having been set, allows a constant flow during the pumping.

(c) The gross continuous sample as collected shall be stirred with the churn-dasher type of stirrer, or mixed by rolling or shaking, and then sampled by means of a thief to get a 1-qt. sample, or more where larger quantities are called for. This method is also applicable to gravity flows where the pipe line is completely filled by the outflowing liquid and has a rising section.

When Materials Flow by Gravity. Materials flowing by gravity through pipe lines which are not completely filled may be sampled by taking dipperfuls^a at the outlet at frequent and regular intervals. The dipper shall have a flared bowl holding approximately 1 quart and a handle of any convenient length. Tinned steel is acceptable. The procedure shall be as follows:

(a) The dipper shall be inserted in the free flowing part of the stream, collecting a sample from the full cross-section of the stream at regular intervals, and the sample collected poured into a clean can of a convenient size, such as 5- or 10-gal. capacity.

(b) If the pumping or delivery rate varies, the samples shall be taken at time intervals, so chosen as to give a sample for equal pumped quantities.

(c) The container into which the individual dipper samples of approximately 1 quart each are poured shall be kept closed and covered, except when the close-fitting lid and cover are lifted to pour in the dipper sample.

(d) The gross amount of material collected should be about 0.1 per cent of the quantity transferred, and the time intervals should be so chosen as to correspond, but not less than 5 gal. nor more than 40 gal. should be collected.

(e) The 5- or 10-gal. can shall be closed and covered, labeled and delivered to the laboratory, where it shall be shaken or mixed with the churn-dasher type of stirrer and portions withdrawn by means of a thief for test.

Sampling at Point of Delivery

Samples may be taken by means of a weighted bottle or can, or from the unloading pipe line as described above. Liquid bituminous materials shall be sampled before heating. Semi-solid or solid bituminous materials shall be rendered fluid by heating. Sampling should be so conducted as to eliminate the possibility of adventitious water resulting from leaky steam heating coils, rain or snow. Samples may be taken from distributors by means of a weighted bottle or can, as described. One sample shall be sufficient.

(a) *Semi-solid or Solid Materials.* Where the lot of material to be sampled is obviously from a single run or batch of the producer, one package or cake shall be selected at random and sampled as described in the following paragraph. Where the lot of material to be sampled is not obviously from a single run or batch of the producer, or where the single sample selected as described above fails on test to conform to the requirements of the specifications, a number of packages or cakes shall be selected at random equivalent to the cube root of the total number of packages or cakes in the lot. For convenience, Table CIV is given, showing the number of samples to be selected for shipments of various sizes:

TABLE CIV
SAMPLING PACKAGES OR CAKES

Packages or Cakes in Shipment	Packages or Cakes Selected	Packages or Cakes in Shipment	Packages or Cakes Selected
2 to 8	2	71 to 343	7
9 to 27	3	344 to 512	8
28 to 64	4	513 to 729	9
65 to 125	5	730 to 1000	10
126 to 216	6	1001 to 1,331	11

Samples shall be taken from at least 3 in. below the surface and at least 3 in. from the side of the container or cake, or from the center of a cake. A clean hatchet may be used if the material is hard enough to shatter and a broad stiff putty knife if the material is soft. An auger, or brace and $\frac{3}{4}$ -in. bit, or other suitable means may also be used. When more than one package or cake in a lot

is sampled, each individual sample shall be not less than $\frac{1}{4}$ lb. in weight. When the lot of material is obviously from a single run or batch of the producer, all samples from the lot shall be melted and thoroughly mixed, and an average sample taken from the combined material for examination. In case more than a single run or batch of the producer is present and the batches can be clearly differentiated, a composite sample shall be prepared for examination from each batch. Where it is not possible to differentiate between the various batches, each sample shall be examined separately.

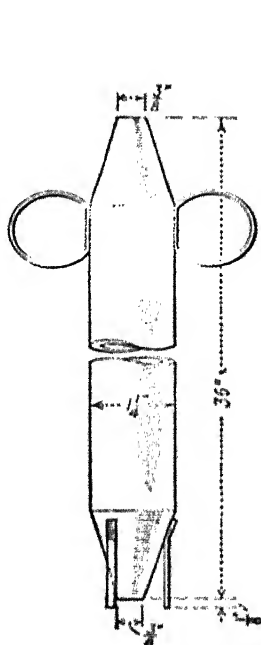
The following method has been suggested for sampling coal-tar pitch from so-called "pitch bays": cut holes to the full depth of the bay, at an agreed upon number of locations. Samples of 8 oz. shall be taken from evenly spaced levels, depending upon the thickness of the pitch layer, as follows:

Up to 1 ft. thick	1 sample from each hole
From 1 to 4 ft. thick	2 " " " "
From 4 to 6 ft. thick	3 " " " "
Over 6 ft. thick	4 " " " "

The samples shall be mixed, crushed and quartered by the method to be described in section (c).

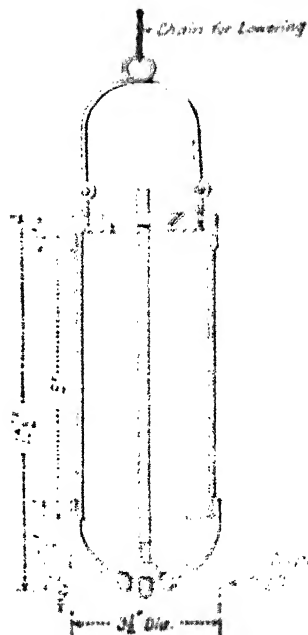
(b) *Liquid Materials.* Samples of liquid bituminous materials from cans, drums or barrels, or tank-cars may be taken by the so-called "thief sampling" method.⁶ For sampling drums, the thief shall be so designed that it will reach within approximately $\frac{1}{8}$ in. of the bottom and should have a capacity of approximately 1 pint or 1 quart. A convenient thief for sampling 50-gal. drum containers is shown in Fig. 215. Three legs, equally spaced around the thief at the lower end, long enough to hold the opening $\frac{1}{8}$ in. from the bottom of the container, aid in securing a good representative sample. Two rings soldered to opposite sides of the tube at the upper end will be found convenient for holding the thief by slipping two fingers through them, leaving the thumb free to close the opening. For sampling cans of 5-gal. capacity or larger, a thief similar to that used for sampling drums but of proportionately smaller dimensions shall be used. For sampling tank-cars, the thief shall be so designed that the sample may be obtained from within

at least 0.5 in. of the bottom. One type of thief suitable for sampling tank-cars is illustrated in Fig. 216, being made from metal tubing and castings, with a valve rod, whose projecting stem strikes the bottom of the car, opening the valve automatically and simultaneously releases the air through the top. Another type of thief



Courtesy A.S.T.M.

FIG. 215.—Thief for Sampling Drums.



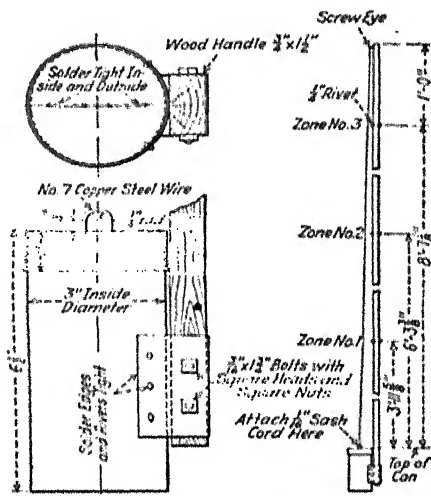
Courtesy A.S.T.M.

FIG. 216.—Thief for Sampling Tank Cars.

designed specially for sampling tank-cars of creosote oil, as illustrated in Fig. 217, consists of a wide-mouthed vessel attached to a wooden rod fitted with a lid that can be withdrawn by means of a cord. This device shall first be lowered until the point marked "Zone No. 1" on the rod is level with the top of the dome of the tank car. The cover of the sampling can shall then be removed, the vessel allowed to fill at that point and then slowly withdrawn.

Extreme care shall be exercised in lifting the rod so as not to disturb the contents of the sampling vessel. Two samples shall be taken in a similar manner with the sampling apparatus lowered to the point marked "Zone No. 2," and one sample shall be likewise taken with the sampling apparatus lowered to the point marked "Zone No. 3." The four samples shall then be combined and mixed at once into a composite sample while the material is thoroughly liquid.

Samples of liquid materials taken from cans, drums or barrels shall be secured by taking 1 qt. of material from packages selected at random according to the cube root method described in section (a). When the lot of material is obviously from a single run or batch of the producer, the samples shall be combined and thoroughly mixed and an average sample taken from the combined material. In case more than one run or batch is present and can be clearly differentiated, a composite sample shall be prepared from each batch. Where it is impossible to differentiate between the various batches or runs, each sample shall be examined separately.



Courtesy A.S.T.M.

FIG. 217.—Zone Sampling Apparatus for Creosote Oil.

(c) *Solid Bituminous Materials in Crushed Fragments or Powder.* Materials such as gilsonite, grahamite, etc., shall be sampled in accordance with methods for sampling coal,⁷ or for less accurate work by the method of "grab sampling."⁸ This method is applicable to all lumpy materials loose in bins, bunkers, barrels, boxes, or contained in sacks. A shovel, if employed for sampling, may be of any convenient size, but should have a width at least twice as great as the maximum dimension of the largest pieces sampled. Grab-sampling is effected by the following general procedure:

Solids are almost always heterogeneous in constitution. It is impossible to give absolutely definite and arbitrary methods for obtaining samples. Each problem must be worked out for itself, bearing in mind the particular conditions.

It is usually preferable to take samples during the unloading of cars or during transit of the material in conveyors. In such instances, a number of small samples should be taken at frequent and regular intervals from the material in transit and these samples combined to form a representative combined sample.

Occasionally, solids are tested as received in bags or barrels. In such instances, it is desirable to take a small sample from a number of packages selected at random and equivalent in number to the cube root (or the next larger whole number), of the total number of packages in the lot.

Generally, samples taken from the bulk, in piles or cars, are unreliable and not representative. Where it is necessary to take such samples before the unloading of the car, small samples should preferably be taken from at least twelve spots throughout the bulk and these small samples collected to form the representative combined sample. In taking such small samples, it is desirable to take eight samples from the corners of the car, four near the bottom and four near the top of the material. To these should be added four samples from the center of the car, two at the top, and two near the bottom of the material.

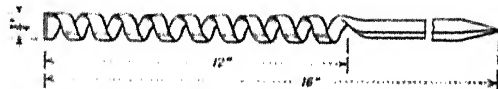
The combined sample taken by any of the above methods should be in amount at least 0.1 per cent of the total bulk of material sampled. These combined samples should be carefully mixed and reduced in size to a convenient laboratory sample, by the standard method of quartering. In carrying out this quartering, a hard clean surface should be selected, free from cracks and protected from rain, snow, wind and beating sun. Cinders, sand, chips from the floor or any other material shall be avoided. The sample shall be protected from loss or gain in moisture, or dust. The combined sample should be carefully mixed, spread out on the surface into a circular layer and divided into four equal quadrants. Two opposite quadrants shall be combined to form the representative reduced sample. If this sample is still too large for laboratory purposes, the quartering operation shall be repeated. In this manner, the sample shall finally be reduced to a size suitable for handling in the laboratory.

For wax and other soft solids in cases, cakes, bags or barrels, when they cannot conveniently be melted and sampled as liquids, a method of "borings sampling" is recommended, as follows:

Use a ship auger which shall be $\frac{3}{4}$ in. in diameter and shall conform to the form and dimensions shown in Fig. 218, and be of such a length as to pass entirely through the material to be sampled.

Opening: Cases and barrels shall have the covers or heads removed. Bags shall have the mouths opened. Cakes shall have the wrappings, if any, opened.

Foreign Matter: If foreign substances, such as dirt, sticks, string, etc., appear upon the surface, they shall be removed. Foreign matter found in the interior shall be included, as it may come, with the borings.



Note: Greater Overall Lengths may be obtained when necessary to pass entirely through the Material to be sampled.

Courtesy A.S.T.M.

FIG. 218.—Auger for Sampling Soft Solids.

Boring Hole Location: Three test holes shall be bored through the body of the material as follows:

One at the center; one at one-quarter of the diameter (or width of the package) from the right side; one at one-quarter of the diameter (or width of the package) from the left side.

If any visible differences appear in the three sets of borings, they shall be preserved, examined and tested separately. If no visible differences appear in the borings they may be combined, preserved, examined and tested as a single sample, but this shall be done only by the laboratory tester after the three sets of borings have been delivered.

Sub-division: If sub-division of the borings is desired, they may be chilled, pulverized if necessary for handling, mixed and quartered till reduced to the amount desired, after their receipt in the laboratory. This sub-division shall not be done in the field by the sampler.

(B) SAMPLING BITUMINOUS PAVING MATERIALS

The special methods given in Table CV are recommended for sampling paving materials.¹⁰

The following procedure has been standardized for sampling paving aggregates:¹¹

Sand, Gravel, Stone, and Slag. Where practicable, samples from commercial sources shall be obtained from the finished prod-

TABLE CV
SAMPLING BITUMINOUS PAVING MATERIALS

Material	Method	Quantity	When Collected	By Whom Tested
Asphalt Sand	Standard methods of sampling stone, slag, gravel, sand and stone block for use as highway materials, including some material survey methods (A.S.T.M. designation: D 75).	5 lb.	Each car or load as it leaves delivery or storage and before mixing. From preliminary or first day's mixtures, from given batch or when source or supply changes or if change occurs in materials received.	Laboratory
			Daily from delivery point, shipments, and sent in care from stock pile.	Plant Laboratory
Screenings	Same as for asphalt sand.	5 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Crushed Stone	Same as for asphalt sand.	15 to 20 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Filler	Composite from eight to ten sacks, mixing and quartering.	5 lb.	Same as for asphalt sand.	Same as for asphalt sand.
Heated Aggregate	By use of shovel as aggregate flows from storage bin.	5 to 20 lb., depending whether source or line aggregate.	At least daily.	Plant Laboratory
Asphalt cement	A. Standard methods of sampling bituminous materials (A.S.T.M. designation: D 149).	1 qt.	Each car or load.	Laboratory
	B. In metal container from valve over asphalt bucket on plant. Mixed and poured into 1 qt. can.	1 qt.	Daily.*	Plant Laboratory and Laboratory
Crude Asphalt	Tentative methods of sampling bituminous materials (A.S.T.M. designation: D 149).	1 qt.	Each car or load.	Laboratory
Refined Asphalt		1 qt.	Each car or load.	Laboratory
Flux		1 qt.	Each car or load.	Laboratory
Finished Mixtures	A. To determine average daily analysis of mixture.	Composite from first 100 of batch.	Daily.	Laboratory
	B. For determination of uniformity of individual or different batches.	From individual batches 4 cu. ft. minimum, for sheet asphalt, 4 lb. minimum, for asphaltic concrete or binder.	As directed.	Laboratory
Asphalt Block	Select blocks representative of production.	2 blocks.	Daily.	Laboratory

*When fluxing at paving plants, preliminary 1 qt. samples shall also be taken from each melting kettle, when ready and completely mixed. This is in addition to daily 1 qt. sample as provided above.

uct. Otherwise the sample shall be taken in accordance with the procedure described. A general inspection of the plant and a record of the screening facilities shall be made. The sample shall preferably be obtained from cars or boats during the loading from stock piles or bins. In order to determine variations in the grading of the material, separate samples shall be obtained at different times while the material is being loaded. If the samples are obtained from a bin, they shall be taken from the entire cross-section of the flow of material as it is being discharged. Approximately 2 to 5 tons of material should be allowed to flow from the bin before the sample is obtained. The testing of separate samples will give a better idea of the variations that occur, but samples shall be mixed and reduced by quartering when the average condition is desired.

Where it is not practicable to visit the plant, samples for both quality and size may be obtained at the destination, preferably while the material is being unloaded. The sampler should realize that segregation of different sizes is very likely to occur, and samples shall be so chosen as to show any differences which occur, both in quality and size of material. Separate samples shall be taken from the top, middle, and bottom of each unit of the shipment. These separate samples shall be mixed to form a composite sample and this sample shall, if necessary, be reduced by quartering, but if information on variation is desired, the separate samples shall be tested. Where test is to be made for size only, it is recommended that tests be made in the field in order not to delay decision on the use of the material. Samples shall also be sent to the laboratory for check tests.

The number of samples required depends on the intended use of the material, the quantity of material involved, and the variations both in quality and size of the aggregate. A sufficient number of samples shall be obtained to cover all variations in the material. It is recommended that each sample of crushed stone, gravel, slag, or sand represents approximately 50 tons of material. Samples of crushed stone, gravel, slag, and sand which are to be subjected to a mechanical analysis in accordance with A.S.T.M. Designation: C 136 shall conform to the weight requirements prescribed in Table CVI.

Bank Run Sand and Gravel. Samples of run of bank (where the sand and gravel are combined) shall weigh at least 100 lb. when the gravel content is 50 per cent or more of the whole. If the gravel is less in percentage, the sample shall be increased in proportion.

NOTE.—Example. When the gravel percentage is 25 per cent of the whole, the sample should weigh 200 lb.

TABLE CVI
SAMPLING MINERAL AGGREGATES^a

Nominal Maximum Size of Particles, Passing Sieve	Minimum Weight of Field Samples, lb.	Minimum Weight of Sample for Test, g.
FINE AGGREGATE		
No. 10	100	1,000
No. 40	100	500
COARSE AGGREGATE		
$\frac{3}{8}$ in.	100	1,000
$\frac{1}{2}$ in.	200	2,000
$\frac{3}{4}$ in.	300	3,000
1 in.	500	5,000
1½ in.	700	7,000
2 in.	900	9,000
2½ in.	1000	10,000
3 in.	1250	12,500
3½ in.	1500	15,000

^a The sample for test shall be obtained from the field sample by quartering or other suitable means to insure a representative portion.

Samples for mechanical analysis shall conform to the requirements for size of sample as prescribed in Table CVI.

Miscellaneous Materials. Samples of slag sand, stone sand, screenings, mine tailings, and all other materials used instead of sand and gravel or broken stone, shall be inspected in the same manner and samples shall be taken in the same manner as prescribed for the materials of similar size and classification.

The following procedure has been standardized for sampling paving brick:¹²

(a) The standard sample for the rattler test shall consist of 10 whole undamaged brick which conform individually to the visual inspection requirements specified. Samples shall preferably be selected at the place of manufacture and tested prior to shipment. However, in special cases where less than 100,000 brick are involved, samples, when required, may be selected subsequent to delivery at the destination.

(b) In general, one standard sample shall be selected for each 15,000 brick or fraction thereof; provided, however, that when the

sampling is done at the place of manufacture prior to shipment and the total number of brick involved is more than 100,000 brick, the number of brick for which one standard sample is considered representative may be increased at the option of the purchaser to not more than 50,000 brick.

Preformed expansion-joint filler should be sampled as follows: ¹³

Each sample shall consist of sufficient material to provide at least 5 test specimens measuring $4\frac{1}{2}$ by $4\frac{1}{2}$ in. in size. One representative sample shall be selected from each shipment of 1000 sq. ft. or fraction thereof, of each thickness ordered.

(C) SAMPLING BITUMINIZED PAPERS AND FABRICS

The following procedure ¹⁴ should be adopted in sampling bituminized roofings and shingles put up in the form of rolls or bundles:

From each shipment or portion thereof representing a product of the same kind, class and weight, a number of rolls or bundles shall be selected at random, equivalent to one-half the cube root of the total number of rolls or bundles included in the lot, except that in lots of 1000 or less, five rolls or bundles shall be taken. If the cube root, as calculated, proves to be a fractional number, it shall be expressed as the nearest higher whole number. For convenience, Table CVII is given, showing the number of samples to be selected from shipments of various sizes:

TABLE CVII
SAMPLING ROOFINGS AND SHINGLES

Packages in Shipment	Number of Packages Selected	Packages in Shipment	Number of Packages Selected
Up to 100	5	8,531 to 8,600	10
101 to 1,000	6	8,601 to 13,648	11
1,229 to 2,744	7	13,649 to 13,824	12
2,745 to 4,096	8	13,825 to 17,576	13
4,097 to 58,12	9	17,577 to 21,952	14

Raw paper and fabricated paper products shall be sampled as follows: ¹⁵

The sample, unless otherwise specified, shall consist when possible of specimens each cut not less than 11 by 11 in. This allows

margin for trimming to exactly 10 by 10 in., which simplifies the calculation in the basis weight determination. A sufficient number of specimens to complete the tests shall be taken. The specimens shall be kept smooth and flat, and protected from exposure to direct sunlight, contact with liquids, and other harmful influences. Care shall be exercised in handling the specimens, if acidity or other chemical characteristics, optical, surface or other physical characteristics affected by the moisture of the hands are to be determined. Specimens to be tested for moisture shall be placed immediately after sampling in an airtight container for storage.

The specimens comprising the sample shall be so selected as to be representative of the entire lot of paper. The units shall be rolls, cases, frames, skids, or bundles. Not less than 5 nor more than 20 sets of specimens, comprising one set from each unit, shall be taken as follows:

Total Units in Shipment	Units to Be Sampled
Less than 1000	5
1000 to 4999	4 to 10 (100)
5000 or more	20

(a) That is, 5 per cent of the total number of units in the shipment

In the case of rolls, care shall be taken to select sheets that are not damaged. It is good practice to discard the first three layers of the roll to be sure of obtaining a unit sample in good representative condition. The specimens shall be cut from sheets taken across the full width of several unharmed layers. In the case of sheet-cut paper, specimens shall be cut from at least five consecutive sheets taken from a point or points over $\frac{1}{2}$ in. from the top or bottom of each case, frame, skid, or bundle. The specimens shall be trimmed with their edges exactly parallel to the machine and cross directions of the paper.

A sufficient number of specimens from each unit sampled shall then be arranged consecutively in rotation to form a representative sample.

In case of necessity for resampling a lot of paper, the samples shall be taken as prescribed, except that they shall be taken from different units than those previously sampled.

NOTE.—Physical tests, except for weight, shall not be made on portions of specimens in which there are flaws or watermarks.

The following method has been standardized for sampling friction tape: ¹⁶

One roll from each 250 or fraction thereof shall be taken at random for test. At least 2 ft. of the outer layers shall be removed and discarded before taking specimen for test.

**(D) SAMPLING BITUMINOUS LACQUERS, CEMENTS, VARNISHES
AND JAPANS; ALSO BITUMINOUS EMULSIONS**

These classes of products shall be sampled as described for "Crude, Refined and Blended Bituminous Substances," "Sampling at Point of Delivery," under Section (b), "Liquid Materials."

The following procedure, however, has been standardized for bituminous cements and plastics:¹⁷

From each shipment the inspector shall take at random a number of packages equivalent to one-half the cube root of the total number of packages in the lot. If this proves to be a fractional number it shall be expressed as the next higher whole number. The contents of each package selected shall be thoroughly stirred until a homogeneous mixture is obtained. One pint of the cement shall then be immediately drawn from each package and transferred to a clean receptacle of a suitable size, which shall be kept tightly covered except while the cement is being introduced. After all the pint samples have been entered, the contents of this container shall be thoroughly stirred and two 1-qt. samples of the composite sample shall be transferred to clean, dry containers, shall immediately be stoppered with new clean corks or well-fitting covers or caps, sealed, and distinctly labeled. One sample shall be transferred to the testing laboratory and the other retained for check analysis in case of dispute.

CHAPTER XXXII

EXAMINATION OF CRUDE, REFINED AND BLENDED BITUMINOUS SUBSTANCES

The present chapter will be devoted to a description of the most important tests used for examining crude, refined and blended bituminous substances. Certain of the tests have been adopted as standards by technical societies, whose committees have been active in this field, accomplishing much to clarify what formerly constituted a veritable jumble of rule-of-thumb methods. Other tests appearing in the current literature will be included where they have been found adequate,¹ but in certain cases these have been amplified or elaborated to conform with the practice followed in the author's laboratory.

The tests which follow are grouped under four headings, viz., physical characteristics, mechanical tests, thermal tests, solubility tests and chemical tests, a section being devoted to each. In general, a test may have one or more objects in view, viz.:

- (1) Serving as a means of identification.²
- (2) Ascertaining the value of the substance for a given use.
- (3) Gauging the uniformity of its supply.
- (4) An aid to factory control in its manufacture, refining or blending, and
- (5) As a criterion of its quality.

The last named may serve as an indication of its purity, the care exercised in its preparation, or its intrinsic value. The tests pertaining to bituminous substances fulfil these requirements as noted in Table CVIII.

Table CIX contains a list of the principal bituminous substances, together with such physical and chemical characteristics as will enable them to be distinguished one from another. Under each heading the minimum and maximum figures are included, based on the author's experience. His intention has been to make the range

TABLE CVIII
TEST REQUIREMENTS

Number	Description	For Purposes of Identification	Adaptability for a Given Purpose	Gauging the Uniformity of Supply	Purposes of Factory Control	As a Criterion of the Quality*
<i>(A) Physical Characteristics</i>						
(Test 1)	Color	YES				
(Test 2)	Homogeneity	YES	Yes	Yes	Yes	YES
(Test 3)	Appearance surface aged one week	Yes	YES			YES
(Test 4)	Fracture	YES				
(Test 5)	Lustre	Yes	YES			YES
(Test 6)	Streak	YES	Yes			
(Test 6a)	Water absorption		YES			Yes
(Test 6b)	Diffusibility		YES			Yes
(Test 7)	Specific gravity	YES		Yes	Yes	YES
(Test 7f)	Viscosity		Yes	Yes	YES	YES
(Test 7g)	Colloidal capacity	YES	YES	YES	Yes	YES
<i>(B) Mechanical Tests</i>						
(Test 8)	Viscosity		YES	Yes	Yes	
(Test 9)	Hardness	YES	YES	YES	YES	
(Test 9d)	Superficiality index	YES	YES	YES	YES	
(Test 10)	Ductility		YES	Yes	Yes	
(Test 11)	Tensile strength (cohesiveness)		YES			
(Test 12)	Adhesiveness		YES			
(Test 12d)	Surface tension	YES	Yes			Yes
<i>(C) Thermal Test</i>						
(Test 12f)	Thermal conductivity	YES	YES			
(Test 12g)	Specific heat	YES	YES			
(Test 12h)	Heat content	YES	YES			
(Test 12i)	Thermal expansion	YES	YES			
(Test 13)	Hardening point	YES	YES	YES	YES	
(Test 14)	Softening point	YES	YES	YES	YES	
(Test 15)	Softening point	YES	YES	YES	YES	
(Test 15g)	Flow point	YES	YES	YES	YES	
(Test 15h)	Expanding point	YES	YES	YES	YES	
(Test 15i)	Twisting point	YES	YES	YES	YES	
(Test 16)	Volatile matter	Yes	YES	YES	YES	Yes
(Test 16a)	Evaporation test	Yes	YES	YES	YES	Yes
(Test 16b)	Distillation test	Yes	YES	YES	YES	Yes
(Test 17)	Flash point		YES	YES	YES	Yes
(Test 18)	Burning point		YES			
(Test 19)	Fixed carbon	YES				
<i>(D) Solubility Tests</i>						
(Test 21)	Soluble in carbon disulfide	YES	YES	YES		YES
(Test 22)	Carbonates	Yes		Yes	Yes	YES
(Test 23)	Soluble in pet.-deum naphtha	YES	Yes	Yes	Yes	
(Test 24)	Insoluble in benzol ("free carbon")	YES	Yes	Yes		
(Test 24a)	Insoluble in other solvents	YES	Yes	Yes		YES
<i>(E) Chemical Tests</i>						
(Test 25)	Water	Yes				YES
(Test 26)	Carbon	YES				
(Test 27)	Hydrogen	YES				
(Test 28)	Sulfur	YES				
(Test 29)	Nitrogen	YES				
(Test 30)	Oxygen (in non mineral matter)	YES				
(Test 30a)	Molecular weight	YES				
(Test 31)	Fat acids	YES	Yes	Yes		
(Test 32)	Naphthalene	YES				
(Test 33)	Sulfonates	YES				
(Test 34)	Sulfonation residue	YES				
(Test 35)	Formic acid reaction	YES				
(Test 36)	Degree of methylation	YES				
(Test 37)	Saponifiable constituents	YES		Yes		YES
(Test 38)	Asphaltic constituents	YES				
(Test 39)	Diazotization reaction	YES		Yes		YES
(Test 40)	Anthracene reaction	YES				
(Test 41)	Leibermann-Storch reaction	YES				

* (a) Purity; (b) care exercised in its preparation; (c) intrinsic value.

TABLE CIX. SYNOPTICAL TABLE OF THE MOST IMPORTANT

Genus	Species	Member	Specific Gravity at 15° of Non-Mineral Matter Test.	Boiler Vacuum at 212° F. 100 ml. Test 34	Penetration at 25° C. Test 36	Spontaneity Index Test 41	Spontaneity of Tests 34 and 41
Bitumens	Petroleums	Non-asphaltic	0.75-0.85		Liquid		0-50
		Semi-asphaltic	0.85-0.95		Liquid		0-50
		Asphaltic	0.95-1.00		Liquid		0-50
	Natural Waxes	Ozokerite	0.85-1.00		5-10	0-50	0-50
		Montan wax	0.90-1.00		0-5	0-50	0-50
Pyrobitumens	Natural Asphalts	Asphalt, mineral matter	1.00-1.10				0-50
		Asphalt, mineral matter	1.10-1.20				0-50
	Asphaltites	Calsonite	1.10-1.20				0-50
		Calsonite patch	1.10-1.20				0-50
		Grahamite	1.10-1.20				0-50
Pyrobitumens	Asphaltic Pyrobitumens	Elatolite	1.10-1.20				0-50
		Wurtzite	1.10-1.20				0-50
		Albitite	1.10-1.20				0-50
		Imperolite	1.10-1.20				0-50
		Asphaltic pyrobitumens, shales	1.10-1.20				0-50
Pyrobitumens	Non-asphaltic Pyrobitumens	Peat oils	0.85-1.00				0-50
		Lignite oils	0.85-1.00				0-50
		Bituminous coal	0.85-1.00				0-50
		Anthracite coal	0.85-1.00				0-50
		Lignite and coal shales	0.85-1.00				0-50
Pyrobitumens	Pyrobitumens Waxes	Wax tars	0.85-0.95	25-30	0-50	0-50	0-50
		Paraffin wax	0.85-0.95	25-30	0-50	0-50	0-50
	Petroleum Tar	Carbonized water tar	0.85-0.95	25-30	0-50	0-50	0-50
		Oil gas tar	0.85-0.95	25-30	0-50	0-50	0-50
		Oil gas tar	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Distillates	Coal Tar	Horizontal gas retort tar	0.85-0.95	25-30	0-50	0-50	0-50
		Inclined gas retort tar	0.85-0.95	25-30	0-50	0-50	0-50
		Vertical gas retort tar	0.85-0.95	25-30	0-50	0-50	0-50
		Coke oven coal tar	0.85-0.95	25-30	0-50	0-50	0-50
		Blas furnace coal tar	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Distillates	Wood Tar	Pine tar	0.85-0.95	25-30	0-50	0-50	0-50
		Hardwood tar	0.85-0.95	25-30	0-50	0-50	0-50
	Miscellaneous Tar	Peat tar	0.85-0.95	25-30	0-50	0-50	0-50
		Lignite tar	0.85-0.95	25-30	0-50	0-50	0-50
		Shale tar	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Residues	Pyrogenous Asphalts	Residual oils	0.85-0.95	25-30	0-50	0-50	0-50
		Blown petroleum asphalt	0.85-0.95	25-30	0-50	0-50	0-50
		Residual asphalt	0.85-0.95	25-30	0-50	0-50	0-50
		Mudg asphalt	0.85-0.95	25-30	0-50	0-50	0-50
		Wurtzite asphalt	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Residues	Petroleum Pitches	Carbonized water gas tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Oil gas tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
	Coal tar Pitches	Horizontal gas retort tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Inclined gas retort tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Vertical gas retort tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Residues	Wood tar Pitches	Coke oven coal tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Blas furnace coal tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Gas producer tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Low temperature tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Pine tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
Pyrogenous Residues	Miscellaneous Pitches	Hardwood tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Resin pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Peat tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Lignite tar pitch	0.85-0.95	25-30	0-50	0-50	0-50
		Shale tar pitch	0.85-0.95	25-30	0-50	0-50	0-50

*R. and B. Method (Test 36) for bitumens, asphaltic pyrobitumens, pyrogenous

Distinguishing Characteristics of Buddhism's Scriptures

[illegible]

waxes and pyrogenous asphalts: Cube Method (Test 154) for tars and pitches.

sufficiently liberal to cover all the commercial varieties, and at the same time prevent the range being too broad, since this would result in unnecessary overlapping. Temperatures will be designated either in degrees Fahrenheit or Centigrade.³

(A) PHYSICAL CHARACTERISTICS

COLOR

Test 1a. Color in Mass. This test is used largely for purposes of identification, and consists in examining a freshly prepared surface of the bituminous material in daylight. The colors range from white, through the various shades of yellow, brown and black. Some possess a greenish or reddish cast, and again others may appear fluorescent. Purified mineral waxes appear pure white, wax tailings a bright yellow, asphalts and pitches are generally brownish-black, grayish-black or black. A method has also been proposed for the color measurement of solid objects.⁴

Test 1b. Color in Solution. This test is likewise of value for purposes of identification and consists in observing the color of the bituminous substance when dissolved in a suitable solvent, either in daylight or when viewed in ultra-violet light.⁵

Method I: Consists⁶ in dissolving 0.25 gram of the substance in 100 ml. of perchlorethylene ($\text{CCl}_2:\text{CCl}_2$), filtering, and pouring into a glass cell, the internal distance between the walls being 0.5 ml. The cell is then placed in a Lovibond Tintometer (B.D.H. pattern) and the color of the solution measured by matching it with standard red, yellow and blue glasses. Typical color values are given in Table CX.

Method II: The Toussaint colorimeter has also been suggested for this purpose,⁷ in which case 0.25 g. of the soluble material is dissolved in 100 ml. benzene and after allowing to stand for 24 hours, examined in 0.25-cm. Lovibond cells. Trinidad epuré asphalt is taken as "standard," as its composition is uniform and the supply is likely to be available for many years to come. Typical color curves are shown in Fig. 219.

For any given type, the harder the asphalt, the darker will be the color. Trinidad asphalt is remarkably light in color for so hard a material. Asphalts recovered from natural rock asphalts by

extraction are all light in color (i.e., they show a comparatively small value of "neutral tint" units in their color). This color test forms a useful guide in ascertaining whether a Trinidad asphalt mixture has been prepared with a flux oil or with a residual petro-

TABLE CX
COLOR VALUES OF ASPHALTS (LOVIBOND TINTOMETER)

In Perchloroethylene	Penetration at 77° F. (Test 96)	Color Value		
		Neutral Tint	Orange	Yellow
Extracted asphalt from:				
St. Jean rock asphalt	79	1.4	6.1	32.3
Sicilian rock asphalt	76	2.4	7.1	40.5
Val de Travers rock-asphalt	73	2.0	8.5	49.5
Trinidad asphalt	11	2.1	6.9	41.0
Residual Petroleum Asphalts:				
From Panuco petroleum	45	3.3	11.0	45.7
From Mexican light petroleum	40	7.2	12.8	49.0
From Venezuelan petroleum	42	7.1	12.9	49.0

In Trichloroethylene	Extracted Asphalt *			Asphaltenes Separated †		
	Yellow	Red	Blue	Yellow	Red	Blue
Trinidad Lake asphalt	1.5	3.1	0.3	5.1	2.0	0.2
St. Jean asphalt (Gard, France)	8.3-9.9	2.2-2.5	0.0	5.2-5.4	1.4-1.6	0.0
Val de Travers asphalt (Swiss)	8.1	2.3	0.0	12.0	3.3	0.0
Verwohle asphalt (Germany)	6.5-8.6	2.2-2.6	0.0	6.6-7.2	2.5-2.7	0.1-0.2
Sicilian asphalt (Ragusa, Italy)	4.8-7.2	1.4-2.3	0.0	5.0-5.1	1.8-1.9	0.0
Buron asphalt (Dutch East Indies)	11.1	3.2	0.0	5.7	2.2	0.0
Pilsonite	20.3	6.6	1.4
Petroleum asphalts (sundry)	4.2-5.1	16.6-25.1	0.2-0.8

* 0.05 g. per 100 ml. trichloroethylene.

† 0.067 g. per 100 ml. trichloroethylene.

leum asphalt, also whether the Trinidad asphalt has been fluxed with a natural rock-asphalt. The colorimetric test has also been proposed for ascertaining the amount of asphalt present in sulfur-bearing ores.*

Method III: Another test* consists in preparing a 0.01 per cent solution of the substance in benzol (freed from mineral constituents), and then titrating an aqueous solution of iodine in potassium iodide (1 gram I_2 and 2 grams KI dissolved in 1000 grams distilled

water) into pure water, in small cylindrical bottles, until its color in daylight exactly matches that of the former. The number of mls. of the iodine solution which contains 1 mg. of iodine is taken as a

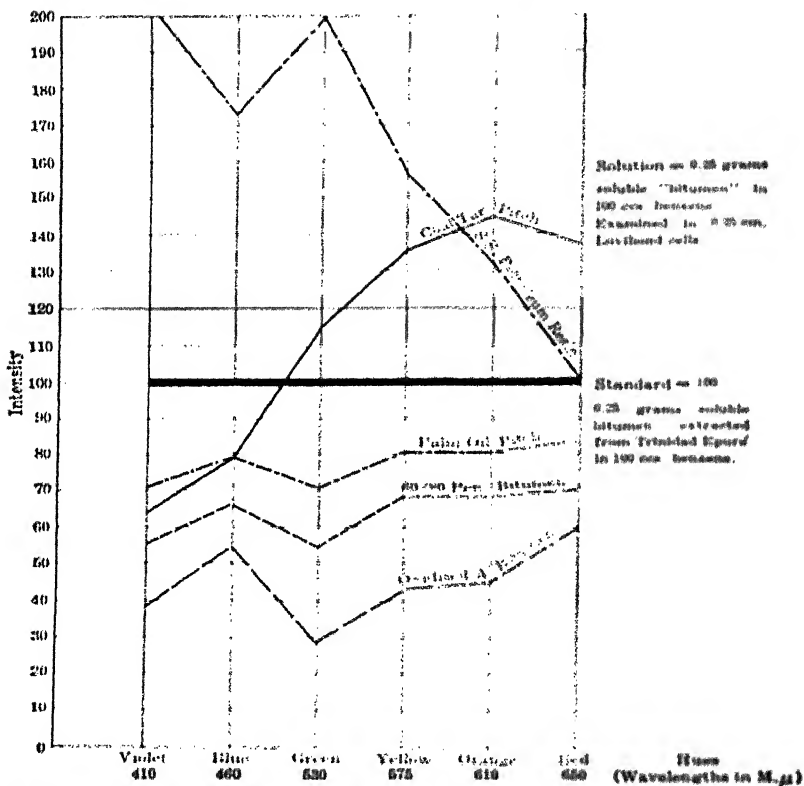


FIG. 219.—Color Values of Bituminous Substances (Toussaint Colorimeter).

measure of the color of the bituminous substance. Representative bituminous substances show a range of from 0.15 to 2.0 ml. iodine solution.

Method IV: Asphalts and tar products when dissolved in solvents may be differentiated by their colors (i.e., fluorescence) when observed in ultra-violet light.¹⁰ Asphalts appear greenish-brown, bitumens greenish-yellow and tar products reddish-brown.

Tar-asphalt mixtures may be compared with color standards of known mixtures and their components estimated to within about 5 per cent.

Method V: With an ultraviolet radiation of 3000-4000 Å, an asphalt content of less than 0.01 per cent may be detected in rocks.¹¹ The luminescence becomes more intense if the rock is first moistened with an organic solvent (e.g., chloroform, petroleum ether or benzol).

Various methods have been standardized for the color measurement of liquids (e.g., refined naphthas, kerosine, etc.).¹²

HOMOGENEITY

This test is used for purposes of identification, for determining the adaptability of the bituminous substance to a given purpose, for gauging the uniformity of supply, for purposes of factory control, as a rough criterion of the purity, and when the bituminous mixture is free from mineral and carbonaceous matter, for ascertaining whether a complete amalgamation of the constituents occur, especially after fluxing.

Test 2a. To the Eye at 77° F. With soft materials this may be ascertained by disturbing a freshly prepared surface of the material (cooled to room temperature) with a rod or spatula, and observing whether any dulling occurs. An alternate method consists in drawing a small pellet into a thread between the fingers, and noting whether it dulls while being drawn out. With hard and brittle substances a freshly fractured surface may be examined. Any evidence of dullness is an indication of: (1) the presence of mineral matter, (2) the presence of free carbon (non-mineral matter insoluble in carbon disulfide), (3) an imperfect blending of the bituminous constituents.

Test 2b. Under Microscope. This is ascertained by spreading a minute quantity of the bituminous material on a microscope slide in a thin layer and examining it by transmitted light under a magnification of 100 to 250 diameters. With hard bituminous materials, the slide should be warmed and the specimen spread uniformly and thinly, while melted. Studies have been made relative to the reactions of films 0.001 in. thick, on glass; on exposure to

air in darkness at various temperatures; on exposure to ultraviolet light; on exposure to oxygen and carbon dioxide in darkness; and on exposure to normal weathering.¹³ The microscopic examination of asphalts manifests the same characteristics as the *petroleum* and in addition, permits the detection of the solid paraffins, which separate from the bituminous matrix in crystal-like masses. Paraffin may be identified positively under a microscope equipped with polariscopic attachment.

The following features have been observed upon examining under a microscope by transmitted light at magnifications of $\times 200$ and $\times 430$, asphalts in films 0.001 in. thick on glass slides, after having been heated to 325° F. up to 278 hours, and at 140° F. up to 2040 hours:¹⁴ coagulation (i.e., curdling or drawing together of the film into nodulated or lace-like patterns), wrinkling, pitting, checking or cracking, hardening (ascertained by scratching with a needle-point), and also crystallization (i.e., formation of waxy constituents into crystals of various shapes and sizes, which become optically active under polarized light).

It is claimed that asphalts may be identified by means of the patterns obtained by etching the surface for 5 seconds with ethyl ether and then examining same under a microscope equipped with a Leitz "Ultrapak" attachment at a magnification of $\times 220$.¹⁵

Test 2c. When Melted. This constitutes a rough test for detecting the presence of substantial amounts of extraneous matter, such as mineral constituents or free carbon. The bituminous material is simply melted and stirred with a rod. If these constituents are present in large quantities, they will impart a gritty feel to the mass, and will often settle out on standing.

Test 2d. Stain Test.¹⁶ *Method 1:* A weight equivalent to 2 ml. is placed in a 25-ml. Erlenmeyer flask, and if it does not flow readily at room temperature, it is cautiously heated over a hot-plate until it flows over the bottom. Upon cooling, add 10.2 ml. of straight-run petroleum naphtha, free from cracked products of any kind, conforming to the following requirements: A.P.I. gravity 49 to 50; initial boiling-point above 300° F.; 50 per cent distills over at 335 to 355° F.; end-point below 410° F.; aniline number (A.S.T.M. D 91-33) 138 to 145° F. Insert a cork stopper in which is fitted an 8-in. length of open ¼-in. glass tubing, and swirl

the contents of the flask with a rapid circular motion for 5 sec., whereupon the flask is immediately immersed to its neck in a bath of gently boiling water. The contents of the flask shall again be swirled for 5 sec. at the end of each minute thereafter until complete dispersion takes place. If the dispersion is not completed in from 6 to 8 min., the test shall be repeated with the bath maintained at a temperature 25° F. lower or higher (glycerin being substituted for the water in the latter case) until dispersion is effected within the stipulated time. After complete dispersion, the flask is allowed to cool to room temperature, and if any loss in weight occurs, it is made up with additional solvent. A drop of the asphalt-solvent mixture is then placed on a No. 50 Whatman filter-paper. If the drop forms a brown or yellowish-brown circular stain, with a darker, solid or annular nucleus in the center, the product is classified as "heterogeneous." If, however, the drop forms a uniformly brown circular stain, judgment shall be reserved, and the solution shall be set aside in its tightly stoppered flask at room temperature in a subdued light, for a period of 24 hr. The mixture shall then be vigorously agitated till uniform, and a drop shall again be placed on the filter-paper. If the drop from the 24-hr. old mixture still forms a uniformly brown circular stain, the product is classified as "homogeneous"; but if a darker, solid or annular nucleus, as described above, now forms in the center of the stain, the product shall be classified as "heterogeneous."

Method II: A quantitative method consists in running the spot test as described in Method I, but instead of employing 10.2 ml. of straight naphtha, exactly the same volume of a predetermined mixture of xylene and standard petroleum naphtha is employed. If the resulting solution, which should be completed within 6 to 8 minutes, tests positive within 24 hr., a fresh solution is prepared, but with a larger proportion of xylene in the mixed solvent, the volume of xylene being progressively increased in increments of 1.0, 0.5, or 0.1 ml. at a time, until a negative test is obtained at the end of 24 hr. The minimum percentage of xylene by volume in the mixed solvent necessary to assure a negative spot for a given asphalt, is reported as the "xylene equivalent" of that asphalt.

Method III: The test has been modified to obtain a numerical index of "homogeneity-hexane-resistance," as follows:¹⁷

(a) *Preparation of Standard Asphalt-naphtha Mixture:*

The asphalt is dissolved in the standard naphtha, as in the foregoing spot test, and then set aside in the dark at approximately 77° F. If negative at the end of 24 hours, there is measured into a clean 25-ml. Erlenmeyer flask 3.9 g. of the asphalt-naphtha mixture (or that quantity that is calculated to contain exactly 4 ml. of the standard naphtha).

(b) *Addition of Hexane Increments:*

From a burette add 0.2 ml. of hexane (starting the stopwatch from zero the moment the addition of the hexane is begun). Immediately thereafter, replace the stopper in the flask, swirl the latter vigorously with a circular motion on the table top for 5 sec., and set aside in a tin or box of approximately the same diameter and height as the flask and lined with black paper to maintain the contents of the flask in subdued light. The entire operation of adding the hexane and swirling should require not more than 15 sec.

(c) *Spot Determination:*

Four and one-half minutes later, or exactly 15 sec. before the expiration of the first 5-min. interval, remove the flask from its black-lined container, swirl vigorously on the table top for exactly 5 sec. as before, remove the stopper, insert the end of a clean glass or metal rod of not over 0.09 in. diameter (cut or ground off square) slightly beneath the surface of the liquid, withdraw it, and let a small drop fall from it upon a No. 50 Whatman filter paper. The size of the drop should be such that it will spread out to not over $\frac{1}{16}$ in. The operation of swirling and depositing a drop on the filter paper should take not over 15 sec., thus completing a full 5-min. cycle.

(d) *Completion of the Test:*

Exactly 5 min. on the stopwatch after beginning the addition of the first increment of hexane, add a fresh increment of 0.2 ml. of hexane, then stopper, swirl, and replace the flask in the black-lined container. At 15 sec. before the expiration of the second 5-min. period, remove and swirl the flask, and withdraw a drop for the spot test exactly as before, in time for a third increment of hexane at the beginning of the third 5-min. period. Continue this procedure till a positive spot is obtained. If the positive nucleus or ring is very faint, additional spots are obtained at the end of at least two more successive 5-min. intervals, to make certain that the faint nucleus or ring first seen becomes progressively more marked in the succeeding spots. Faint nuclei or rings that may appear in

sporadic spots but do not reappear in subsequent ones, or remain very faint and vaguely defined, are ignored as not authentic or significant.

(c) *Reporting Hexane Resistance:*

The "hexane resistance"—that is, the resistance of the asphalt-naphtha mixture to the flocculating force of the hexane—is considered equivalent to the total number of hexane increments required to give the first *authentic* positive spot, however faint.

The foregoing test will serve to detect an unstable internal phase relationship (i.e., heterogeneity) in petroleum asphalts and their mixtures, caused by:

- (1) The formation of compounds by cracking or overheating.
- (2) Incompatible waxy bodies.
- (3) Products formed during exposure to the weather.
- (4) The presence of sludge asphalts, coal-tar pitch, etc.
- (5) Faulty synthesis or blending of asphaltic mixtures.

Method IV: Further modifications of the "stain test" involve:¹⁸

Increased Standing Time. In which standard naphtha is used, as in the original test, and the mixture is then allowed to stand, and the number of days noted for a positive spot to appear.

Degree of Naphtha Dilution. In which standard naphtha is used, as in the original test, and the volume increased until a positive spot test results, and the naphtha-asphalt ratio recorded.

The results given in Table CXI have been obtained with typical asphalts, when subjected to the "heterogeneity test" described above.

APPEARANCE SURFACE WHEN AGED

Test 3. Appearance Surface Aged Indoors One Week. A small quantity of the bituminous material is carefully melted at the lowest possible temperature and poured into a tin ointment box or deep seamless can as used for determining the volatile matter (Test 16). The surface should be free from froth or bubbles and allowed to cool in a place free from draughts. When cool, the surface is examined, and then covered to protect it from dust. At the end of a week the cover is removed and the surface re-examined. If bright and lustrous, it will indicate a perfect amalgamation of the

constituents, also the absence of oily, greasy and undissolved constituents. A lustreless surface is an indication of the presence of extraneous mineral or carbonaceous matter, or evidence that the constituents do not blend or amalgamate properly. If the surface appears greasy or wax-like, vaseline- or paraffin-like bodies are present, since these have the property of separating or "sweating" from the bituminous matrix on standing. This would prove objectionable where the bituminous material is to be used for surfacing prepared roofings dusted with talc, or for manufacturing bituminous paints, varnishes or japans. This test is accordingly used for purposes of identification, determining the adaptability of the substance for a given purpose and as a criterion of its quality.

Test 3a. Exudation or "Bleeding" Tendency. This test is used for testing the extent of incompatibility between the asphalts used as saturant and surface-coating of prepared roofings and shingles, as evidenced by the appearance of dull-black spots, or unsightly discoloration of the talc or granular surfacing, that may develop during aging in storage. The following procedure has been proposed rapidly to detect any such strike-through tendency: ¹⁹

The coating is warmed to a fluid condition. It should then be poured into the lid of a 3-ounce (88.7-ml.) penetration tin or other convenient receptacle in a layer 0.3 to 0.6 cm. (0.125 to 0.25 in.) thick. To remove air bubbles, the surface of the coating may be momentarily heated. The surface area and total weight of the specimen are determined and the surface is then given a preliminary dusting with fine roofer's talc, evenly distributed over the surface, neither the surface nor the talc being handled by the fingers during this operation. The excess of nonadherent powder is removed by inverting the specimen and allowing the container to drop 2.5 cm. (1 in.) onto the table top. A second application of fine talc is then made by gently shaking or tapping a 300-mesh sieve held 7.5 cm. (3 in.) above the surface of the specimen, so that a fine mist rather than agglomerated particles of the powder will accumulate on the specimen. This operation is continued with occasional weighings until a uniform film of talc weighing 0.025 g. per sq. in. (6.45 sq. cm.) has been obtained. Uniformity in the thickness of the talc film is of great importance in obtaining reproducible results, for the thicker the layer of talc (up to a certain limit), the wider will be the ring formed.

A drop of the saturant about 0.16 cm. (0.0625 in.) in diameter is placed upon the talc-dusted surface of the coating. This may be

done most conveniently by plunging the end of a heated spatula or paring knife into the cold saturant and, after the excess has drained off, allowing a drop of suitable size to fall on the dusted surface from a height of about 1.25 cm. (0.5 in.). Several drops of the same or different saturants may be applied to a single specimen of dusted coating.

The specimen is then placed in an oven maintained at a temperature of $43.33^{\circ} \pm 2.8^{\circ} \text{C.}$ ($110^{\circ} \pm 5^{\circ} \text{F.}$) for a period of 72 hours. The average width of the dark-brown or black ring of discolored talc that has formed at the end of 72 hours around the periphery of the spot is determined to the closest 0.1 mm. by means of a scale of suitable dimensions and a good magnifying glass. This dark ring is usually sharply defined, and the vague penumbra that sometimes develops beyond the area of marked discoloration should be disregarded. A roughly quantitative estimate of the degree of bleeding to be anticipated in roofing in which any two asphalts are to be used, may be based on the width of ring of discolored talc that they develop in the exudation test. If no ring whatever is formed in that test, not the least traces of bleeding will occur in the roofing made with the two asphalts.

FRACTURE

Test 4. Conventional Method. This is ascertained upon cleaving the specimen by subjecting it to a sharp blow, and examining the cleavage surface. Only hard and "brittle" bituminous substances will yield to this test, including the hard asphalts and asphaltites. The fracture may either appear conchoidal (rounded and curved like a shell), or hackly (jagged, irregularly and rough).

LUSTRE

Test 5. Conventional Method. Method I: This indicates the way light is reflected from a freshly fractured surface, which may be bright or vitreous—indicating that it has the brilliancy or shine of glass; greasy—indicating that it presents an oily or greasy surface; waxy—indicating that it has the characteristic appearance of wax; or dull—indicating that the surface is without lustre. These manifestations are used for purposes of identification, and for determining the adaptability of the bituminous material for manufacturing lacquers, varnishes and japans.

Method II: A photoelectric device for measuring the gloss

quantitatively, as illustrated in Fig. 220.²⁰ The asphalt is melted and poured into a shallow glass tray which is cooled in air or water at 77° F. for 24 hours; then laid on a flat surface under the gloss-meter and the legs adjusted for height until the deflection of the

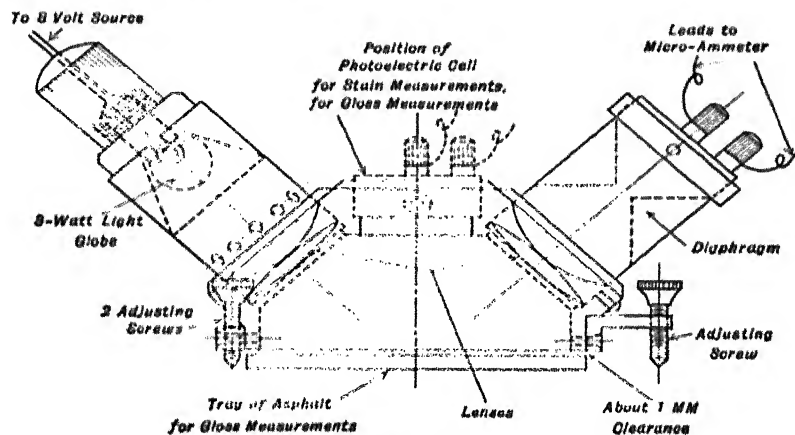


FIG. 220.—Diagram of Glossmeter.

microammeter is at the maximum. In the case of the asphalt whose gloss has been chosen as the standard (e.g., a 40-50 penetration Mexican residual asphalt), the deflection is adjusted to correspond to 100 scale divisions. Hence the greatest deflection produced by the sample under test will indicate the percentage of gloss.

TABLE CXII

COMPARISON OF GLOSSMETER MEASUREMENTS WITH VISUAL CLASSIFICATION AND WAX CONTENT ON FRACTIONS FROM RESIDUAL ASPHALT

Cut No.	Penetration at 25° C. (77° F.)	Softening Point (Ring-and-Ball) ° C.	Softening Point ° F.	Saybolt Furol Viscosity at 98.9° C. (210° F.)	Wax (Hofmeier Method) Wt. %	Gloss Measurements %	Visual Appearance
1	2	80.6	177	...	1.16	107	Bright
2	20	56.1	133	...	1.61	95	Bright
3	230	36.7	98	378	2.24	90	Bright
4	> 300	151	2.69	89	Bright
5	> 300	82	3.00	85	Slightly dull
6	> 300	54	4.85	84	Dull
7	> 300	39	4.93	81	Dull

Table CXII gives the results obtained on a number of fractionated cuts from a residual asphalt, and Table CXIII shows the use of the glossmeter for determining the effectiveness in removing wax from a residual asphalt. The instrument may also be employed for measuring the staining characteristics of asphalts (see Test 2d).

TABLE CXIII

SURFACE GLOSS AS AN INDICATION OF EFFECTIVENESS OF PROCESSING

Cut No.	Untreated Stock		First Processing		Second Processing	
	Gloss Measurement %	Visual Appearance	Gloss Measurement %	Visual Appearance	Gloss Measurement %	Visual Appearance
1	91	Hazy	103	Bright	111	Bright
2	84	Hazy	101	Bright	111	Bright
3	79	Dull	90	Hazy	101	Bright
4	77	Dull	84	Hazy	88	Hazy
5	67	Dull	80	Dull	85	Hazy
6	66	Dull	74	Dull	78	Dull
7	62	Dull	72	Dull	77	Dull
8	57	Dull	67	Dull

STREAK

Test 6. Conventional Method. This represents the color of the powder which is left behind on drawing a piece of the solid bituminous material across the surface of unglazed porcelain. Hard bituminous materials only will yield to this test. The streak may be classified as white (where no streak is visible), yellowish, yellowish-brown, brown, brownish-black and black. This test is of value for purposes of identification, and as an indication of the suitability of the substance for use with colored pigments.

WATER ABSORPTION

Test 6a. Quantitative Method.²¹ The capacity of bituminous substances to absorb water may be ascertained by the following method: clean brass plates measuring 3 by 4 by $\frac{1}{32}$ in. (of which the surface area measures 21 sq. in., excluding the edges) are heated to 250° F. and dipped into the melted asphalt to a depth of $3\frac{1}{2}$ in., so as to give a film weighing 7.5–8.0 grams in one dip. Each plate is then heated carefully over a burner to expel the air from the substance and to seal the edges. Weigh and immerse in

distilled water at room temperature for 52 weeks. Remove the plates, dry the surface quickly with a sheet of filter paper and re-weigh. The asphalts specified in Table CXIV were examined in

TABLE CXIV
WATER-ABSORPTION TEST ON BLOWN PETROLEUM ASPHALTS

		(1) Blown Mid-Continental Asphalt	(2) Blown Mexican Asphalt	(3) Blown Venezuelan Asphalt	(4) Blown West-Texas Asphalt	(5) Blown Colombian Asphalt
Test 7)	Specific gravity at 77° F.	1.006	1.055	1.037	1.026	1.099
Test 9b)	Penetration at 115° F.	26	17½	30	16	26
	Penetration at 77° F.	17	10	13	9½	17
	Penetration at 52° F.	8½	6	7	5½	10
(Test 15b)	Fusing point (R. & B.).	230° F.	245° F.	234° F.	221° F.	230° F.
(Test 19)	Fixed carbon.	16.16%	19.48%	19.00%	17.15%	15.95%
(Test 38c)	Asphaltenes.	31.6%	46.2%	40.2%	38.4%	36.2%
(Test 38d)	Asphaltic resins.	25.5%	16.0%	17.1%	20.9%	15.0%
(Test 38e)	Oily constituents.	39.1%	37.0%	41.7%	40.0%	47.2%

this manner. In this test blown asphalts derived from petroleum showed increases in weight ranging from 1.5 per cent to 12.0 per cent (Colombian petroleum asphalt the lowest and mid-continental

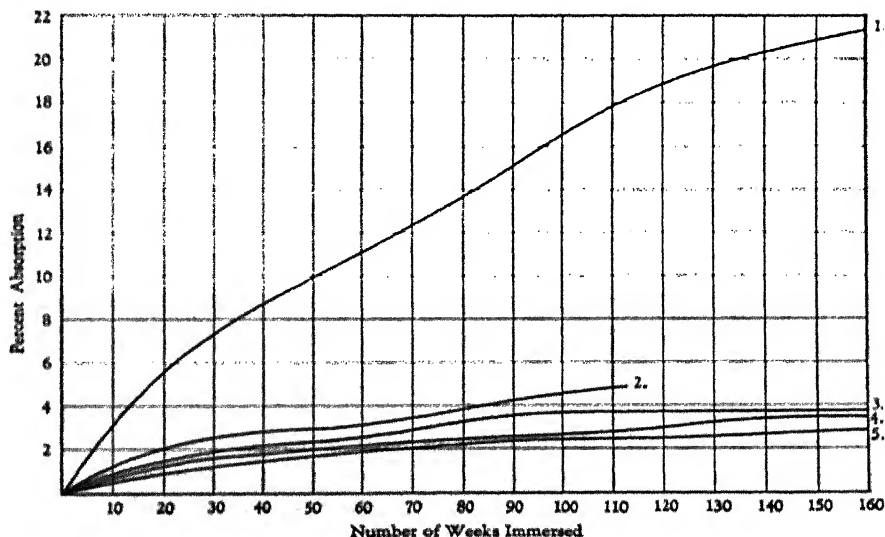


FIG. 221. Water-absorption of Blown Petroleum Asphalts (see Table CXIV for Identity).

asphalt the highest). At the end of 156 weeks, the increases in weight ranged from 2.8 per cent to 21.3 per cent respectively, as illustrated in Fig. 221.

DIFFUSIBILITY

Test 6b. Water Diffusibility. *Method 1:* The "diffusibility constant" is the number of grams of water vapor which will diffuse through a 1 cm. cube of the substance in 1 hour under a vapor pressure difference of 1 mm. mercury at 77° F.²² The bituminous substance is mounted on an aluminum cell of the form illustrated in Fig. 222. The cell is placed in a desiccator maintained at 77° F., with a drying agent attached to a calibrated spring, which serves to give direct readings of the weight of water absorbed. Water passes from the cup at the bottom, through the diaphragm, and into the space above, where it is absorbed by magnesium perchlorate held on the aluminum foil pan supported by the calibrated fused quartz spring. With this cell, small amounts of water are easily detected without disturbing the system.

A solution for maintaining constant humidity was placed in the cup, an asphalt diaphragm sealed to an accurately turned and ground flat brass ring was placed over the mouth of the cup, and over this the inverted test tube containing the quartz spring supporting some anhydrous was sealed with a thick layer of paraffin. The assembled cells were placed in a thermostatically controlled oven maintained within 0.2° C. of the desired temperature. The elongations of the springs were measured with a cathetometer without removing the cells from the thermostat.

In some cases the absorption of the water vapor initially present in the inverted test tube resulted in a pressure differential across the diaphragm sufficient to cause it to warp or fracture. To avoid this, a capillary tube was pulled out on the side of the cell. After assembly of the cells, the capillaries were left open for 3 or 4 hours and then sealed. In all cases where this procedure was followed, there was no damage to the asphalt membranes as a result of pressure reduction.

The quartz springs were wound on a device which consisted of

a rotating quartz mandrel with an automatic screw feed from which a quartz thread was fed through an oxygen-gas flame. The diame

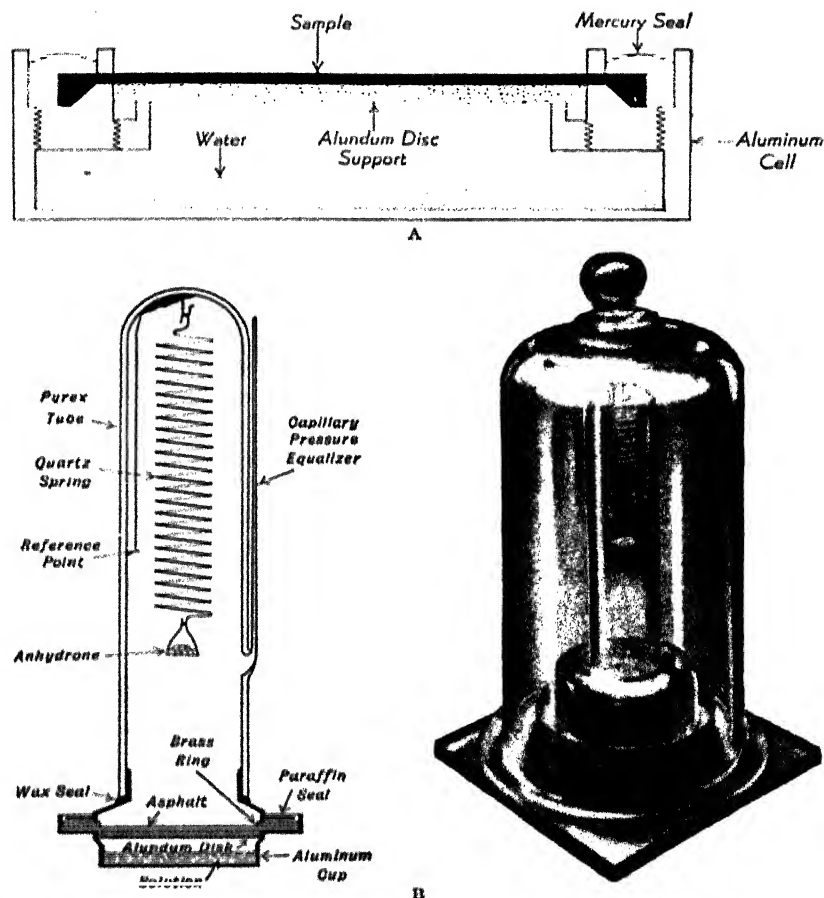


FIG. 222.—Apparatus for Ascertaining Diffusibility. A—Cross-section of Cell; B—Apparatus Assembled.

ter of the quartz thread and the diameter of the spring were correlated to give the desired force constants in the springs. These ranged from 0.5 to 1.5 mg. per mm. deflection. Calibration of

the springs showed that in all cases the deflection was directly proportional to the load.

The membranes were prepared by first evacuating the molten bitumen. The material was heated to about 50°C. above its A.S.T.M. melting point and evacuated with a water aspirator for about 30 min., which was usually sufficient time to eliminate voids in the membranes. After adequate evacuation, a small amount of the material was placed between carefully ground, flat, amalgamated brass plates, and the assembly placed in an oven with the temperature maintained near the melting point of the material. Spring loading of the upper brass plate caused the material to spread until the plate rested upon spacers placed on the lower plate, which regulated the thickness of the membrane. The disk for use in the diffusion cells was cut from each membrane with a circular, amalgamated, brass knife edge at temperatures suitable to the bitumen being cut. In this manner membranes were obtained which did not fail when subjected to a spark gap test. In the case of very thin membranes, examination with a strong light failed to disclose any imperfections. The thickness was determined with a micrometer and also from the weight, area, and density of the material. Ball-and-ring softening points of the evacuated materials were found to be substantially the same as for the original materials.

Method II: Low melting point materials were too soft to be dealt with by the procedure just described. In these cases the customary diaphragm was replaced by a perforated brass disk, the holes of which were filled with the petroleum or asphalt. If the holes were not too large, the material was supported by its surface tension and voids did not develop. The plates used had an area of 18.6 sq. cm. and had 100 holes per sq. cm. drilled with a B. and S. gage 68 drill. The voids constituted about 47 per cent of the cross section of the disks.

To prevent the condensation of water upon the under side of the membranes, the humidity in the air space was kept below 100 per cent by the use of saturated solutions in place of pure water, as mentioned above. This procedure not only prevented condensation but also permitted an investigation of the dependence of the diffusion constant upon the pressure gradient.

The apparatus was maintained at constant temperature ($25 \pm 0.2^\circ$ or $35 \pm 0.2^\circ$ C.) in an air-thermostat equipped with glass doors so that the deflections of the quartz springs could be measured with a cathetometer without disturbing the apparatus. From twelve to twenty deflection measurements were made on the quartz spring for each membrane.

The measurements are ordinarily expressed as water permeability constants obtained by Fick's diffusion law:

$$W' = \frac{K.A.P.T}{L}$$

where W'	weight of water diffused, g.
A	area of membrane, sq. cm.
L	thickness of membrane, cm.
P	vapor pressure differential, mm. Hg
T	time of diffusion, hr.
K	permeability constant

Some typical results of this test are cited in Table CXV.

TABLE CXV

PERMEABILITY OF BITUMENS TO WATER AT 25° C. (77° F.)

Bitumen	Penetration at 25° C.	Specific Gravity	Permeability Constant, (G.) (Cm.) / (Sq. Cm.) (Mm.) (Hr.) $\times 10^9$
Air-blown asphalt	16	1.026	9.0
Air-blown asphalt	5	1.031	6.8
Steam-refined asphalt	15	1.030	6.0
Steam-refined asphalt	5	1.036	4.1
Coal-tar pitch	16	1.17	7.0
Plasticized pitch (containing about 35% inorganic filler)	4	1.36	2.8

Test 6c. Air Permeability. A thin layer (0.001 in.) is applied to the outside of a porous cement pipe and subjected to air pressure inside the pipe, ranging from 765 to 2760 mm. mercury at a predetermined temperature. The extent of passage of the air through the coating is measured.²⁸

SPECIFIC GRAVITY

This test is of value: (1) in identifying bituminous materials; (2) for controlling the uniformity of supply; (3) for purposes of factory control; (4) for figuring the weight of a given volume as delivered in tank cars, when stored in tanks, or else upon filling into containers; (5) for calculating the volume of the bituminous binder in pavements. The specific gravity is of special value when considered in connection with the fusing-point or hardness.

The following terms have been defined^a relating to specific gravity:

(A) *Absolute Specific Gravity* (of solids and liquids). The ratio of the weight referred to vacuum of a given volume of the material at a stated temperature to the weight referred to vacuum of an equal volume of gas-free distilled water * at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

$$\text{Absolute Specific Gravity } x''/x^{\circ} \text{ C.} \quad ,$$

where x is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

$$\text{Absolute Specific Gravity } x''/y^{\circ} \text{ C.} \quad ,$$

where x is the temperature of the material and y the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

(B) *Specific Gravity* (of solids and liquids). The ratio of the weight in air of a given volume of the material at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

$$\text{Specific Gravity } x''/x^{\circ} \text{ C.} \quad ,$$

where x is the temperature of the material and the water.

* Distilled water boiled vigorously in vacuum.

(b) When the temperatures of the material and of the water are *not the same*:

Specific Gravity x°/y° C.

where x is the temperature of the material and y is the temperature of the water.

NOTE.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

(C) *Apparent Specific Gravity* (of solids). The ratio of the weight in air of a given volume of the impermeable portion of a permeable material (that is, the solid matter including its impermeable pores or voids) at a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

Apparent Specific Gravity x°/x° C.

where x is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

Apparent Specific Gravity x°/y° C.

where x is the temperature of the material and y is the temperature of the water.

NOTE 1.—In scientific circles, specific gravity and density determinations made in air (that is, uncorrected to vacuum) are frequently distinguished by the adjective "apparent." Thus, the specific gravity defined by definition B would be designated as "apparent specific gravity" and that defined by definition A as "specific gravity." But in industry, the terminology is more generally in accordance with that given in these definitions.

NOTE 2.—The terms "permeable" and "impermeable" cannot be rigidly defined for general application. The exact meaning in a particular application is the conventional one inferred by the procedure specified for determining the specific gravity of the material in question.

NOTE 3.—In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

(D) *Bulk Specific Gravity* (of solids). The ratio of the weight in air of a given volume of a permeable material (including both permeable and impermeable voids normal to the material) at

a stated temperature to the weight in air of an equal volume of distilled water at a stated temperature. It shall be stated thus:

(a) When the temperatures of the material and of the water are the *same*:

$$\text{Bulk Specific Gravity } x^{\circ}/x^{\circ} \text{ C.} \quad ,$$

where x is the temperature of the material and the water.

(b) When the temperatures of the material and of the water are *not the same*:

$$\text{Bulk Specific Gravity } x^{\circ}/y^{\circ} \text{ C.} \quad ,$$

where x is the temperature of the material and y is the temperature of the water.

NOTE 1. See Note 2 under (C) "Apparent Specific Gravity."

NOTE 2. In the interest of standardization and simplification, the first form of statement of specific gravity (a) should be employed wherever practicable.

Density, i.e., mass per unit volume, is a much more fundamental concept than specific gravity, and there is an increasing tendency to employ density instead of specific gravity, both in scientific and in industrial work.²⁰ Densities are generally most conveniently expressed in terms of grams per milliliter at t° C., designated by the symbol $\rho_{t^{\circ}\text{C.}}$. The following formula is used for converting "specific gravity" into "density":

$$\rho_{t_1} = S_{t_1/t_2} \times \rho_{t_2}$$

where ρ_{t_1} = the density of the substance at the temperature,

ρ_{t_2} = the density of water at the temperature,

S_{t_1/t_2} = the specific gravity of the substance at t_1° compared with water at t_2° .

Hence:

$$\rho_{15.5^{\circ}\text{C.}} = S_{15.5^{\circ}\text{C./15.5^{\circ}\text{C.}} \times 0.99905$$

$$\rho_{20^{\circ}\text{C.}} = S_{20^{\circ}\text{C./20^{\circ}\text{C.}} \times 0.99823$$

$$\rho_{25^{\circ}\text{C.}} = S_{25^{\circ}\text{C./25^{\circ}\text{C.}} \times 0.99707$$

$$\rho_{30^{\circ}\text{C.}} = S_{30^{\circ}\text{C./30^{\circ}\text{C.}} \times 0.99599$$

Table CXVI, based upon the foregoing relations, gives the corrections, expressed as units in the fourth decimal place, to be subtracted from the specific gravity S (t° C./ t° C.) in order to obtain the corresponding density in grams per milliliter at t° C.:

TABLE CXVI

CONVERSION OF SPECIFIC GRAVITY TO DENSITY

Sps. Gr. (S^1)	From $S_{0^\circ \text{ C.}/10^\circ \text{ C.}}$ to $\rho_{10^\circ \text{ C.}}$	From $S_{15.5^\circ \text{ C.}/15.5^\circ \text{ C.}}$ to $\rho_{15.5^\circ \text{ C.}}$	From $S_{38^\circ \text{ C.}/20^\circ \text{ C.}}$ to $\rho_{38^\circ \text{ C.}}$	From $S_{25^\circ \text{ C.}/25^\circ \text{ C.}}$ to $\rho_{25^\circ \text{ C.}}$	From $S_{38^\circ \text{ C.}/38^\circ \text{ C.}}$ to $\rho_{38^\circ \text{ C.}}$
0.60	-1	6	-11	-18	-42
0.65	-1	6	-12	-19	-45
0.70	-1	-7	-12	-20	-49
0.75	-1	-7	-13	-21	-52
0.80	-1	-8	-14	-22	-56
0.85	-1	-8	-15	-25	-59
0.90	-1	-9	-16	-26	-63
0.95	-1	-9	-17	-27	-67
1.00	-1	-10	-18	-29	-70
1.05	-1	-10	-19	-31	-74
1.10	-1	-10	-19	-32	-77
1.15	-1	-11	-20	-34	-80
1.20	-2	-11	-21	-35	-84
1.25	-2	-12	-22	-37	-87
1.30	-2	-12	-23	-38	-91

Example. The density at 25° C. in g/ml of a liquid having a specific gravity $S_{25^\circ \text{ C.}/25^\circ \text{ C.}}$ of 0.8564 is obtained as follows:

The correction given in the table for $S_{25^\circ \text{ C.}/25^\circ \text{ C.}}$ of 0.85 is 25, and for $S_{28^\circ \text{ C.}/25^\circ \text{ C.}}$ of 0.90 is 26, and therefore, by interpolation, the correction for $S_{25^\circ \text{ C.}/25^\circ \text{ C.}}$ of 0.8564 is 256. Hence, the density at 25° C. of a liquid having a specific gravity $S_{25^\circ \text{ C.}/25^\circ \text{ C.}}$ of 0.8564 is $(0.8564 - 0.00256)$ g/ml, i.e. 0.8538 g/ml.

When specific gravity is required with accuracy in the fourth place of decimals, and is determined by a weighing method (bottle, pycnometer, etc.) it is necessary to apply corrections for the buoyancy effect of the air as follows:

If W_w be the observed weight in air of the water content at 15.5° C. , and W_s be the observed weight in air of the sample contained in the bottle (or pycnometer, etc.) at 15.5° C. ; then the approximate specific gravity $S^1_{15.5^\circ \text{ C.}/15.5^\circ \text{ C.}}$ is given by the equation:

$$S^1_{15.5^\circ \text{ C.}/15.5^\circ \text{ C.}} = \frac{W_s}{W_w}$$

The value given by the above formula may be corrected for the buoyancy effect of the air by the figures given in Table CXVII expressed as units in the fourth decimal place, to be added to (when the sign is positive) or subtracted from (when negative) the approximate specific gravity $S_{17.6^\circ\text{C./15.5}^\circ\text{C.}}$ to give the corrected specific gravity.

TABLE CXVII
CORRECTION FOR BUOYANCY AND CHANGE IN VOLUME OF BOTTLE

$S_{17.6^\circ\text{C./15.5}^\circ\text{C.}}$	15.5° C.	20° C.	25° C.	30° C.
0.60	+5	+4	+3	+1
0.65	+4	+3	+3	0
0.70	+4	+3	+2	-1
0.75	+3	+2	+1	-1
0.80	+2	+1	0	-2
0.85	+2	+1	-1	-3
0.90	+1	0	-1	-4
0.95	+1	-1	-2	-5
1.00	0	-1	-3	-6
1.05	-1	-2	-3	-7
1.10	-1	-2	-4	-8
1.15	-2	-3	-5	-9
1.20	-2	-4	-6	-9
1.25	-3	-4	-6	-10
1.30	-4	-5	-7	-11

Example.—Take the case where:

W_1 equals the weight water to fill bottle at $15.5^\circ\text{C.} = 49.892\text{ g.}$

W_2 equals the weight substance to fill bottle at $17.6^\circ\text{C.} = 43.271\text{ g.}$

$$S_{17.6^\circ\text{C./15.5}^\circ\text{C.}} = W_2/W_1 = 43.271/49.892 = 0.8673$$

From Table CXVII the correction for $S_{17.6^\circ\text{C./15.5}^\circ\text{C.}}$ is $+0.0001$. Hence the specific gravity $S_{17.6^\circ\text{C./15.5}^\circ\text{C.}}$ of the substance is $0.8673 + 0.0001$, or 0.8674 .

In the United States, it is customary to ascertain the specific gravity of bituminous materials at $77^\circ\text{F./77}^\circ\text{F.}$, petroleum products at $60^\circ\text{F./60}^\circ\text{F.}$, and creosote oils at $100^\circ\text{F./60}^\circ\text{F.}$

For converting the specific gravity of a substance found at a

higher temperature to the standard temperature (lower), the following formula should be used: ²⁶

$$\text{Sp. gr. Substance at } t_1/t_1 = \text{Sp. gr. Substance at } t_2/t_1 + k(t_2 - t_1)$$

in which t_2 = the temperature at which the specific gravity of the substance was determined,

t_1 = the temperature (lower) at which the specific gravity of the substance is to be calculated, and

k = the coefficient of cubical expansion per deg. F., which is constant for the particular substance.

Test 7a. Hydrometer Method (Used for Thin Fluid Substances). Where speed is essential and great accuracy not required, the specific gravity of fluid bituminous materials may be determined with a hydrometer having its scale sub-divided to unity in the third place of decimals. Usually a series of hydrometers are used, ranging respectively from 0.800 to 0.900, 0.900 to 1.000, 1.000 to 1.080, 1.070 to 1.150, 1.150 to 1.230, and in such dimensions as to enable them to be used in a 100 ml. cylinder approximately 300 mm. long having an inside diameter of not less than 32 mm. as illustrated in Fig. 223. The hydrometer shall conform to the following requirements as to dimensions:

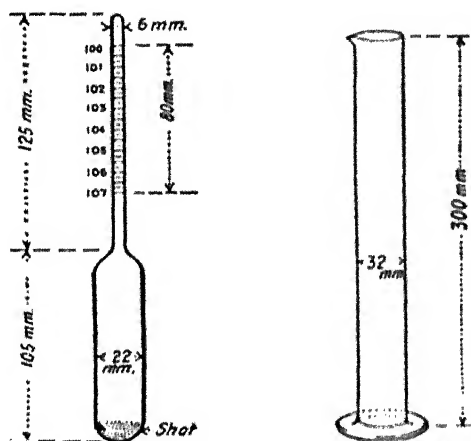
	Dimension	Permissible Variation
Length of stem	125 mm.	6 mm.
Length of bulb	105 mm.	5 mm.
Length of scale	80 mm.	4 mm.
Diameter of stem	6 mm.	0.5 mm.
Diameter of bulb	22 mm.	1 mm.

Method I: For testing crude petroleum and its products the following procedure has been standardized: ²⁷

The sample to be tested for gravity shall be poured into the clean hydrometer cylinder without splashing, so as to avoid the formation of air bubbles and to reduce to a minimum the evaporation of the lower boiling constituents of the lighter oils. If air bubbles are formed they shall be removed after they have collected on the surface, by touching them with a piece of clean blotting paper or filter paper before the hydrometer is placed in the sample.

The cylinder containing the prepared sample shall be placed in a vertical position in a place free from air currents. The hydrometer shall be carefully lowered into the sample to a level two smallest

scale divisions below that at which it will float and shall then be released. When the hydrometer has come to rest, floating freely away from the walls of the cylinder, the gravity shall be read as the point at which the surface of the sample apparently cuts the hydrometer scale. In the case of samples sufficiently transparent this point shall be determined by placing the eye slightly below the level of the liquid and slowly raising it until the surface of the sample first seen as a distorted ellipse seems to become a straight line



Courtesy A. S. T. M.

FIG. 223.—Hydrometer for Liquid Bituminous Substances.

cutting the hydrometer scale. In the case of nontransparent samples where this procedure cannot be followed, the point on the hydrometer scale to which the sample rises above the main surface of the liquid shall be read with the eye placed slightly above the plane of the surface of the sample. This reading shall then be corrected, by subtracting in the case of A.P.I. hydrometers, or adding in the case of specific-gravity hydrometers, an amount equal to the height which the sample rises on the hydrometer stem above the main liquid surface. This height will vary for different oils and different hydrometers and the amount of correction necessary will depend upon the width of the hydrometer scale graduations. The necessary correction factor shall be determined, therefore, for the particular hydrometer in use by observing the height above the main surface of the liquid to which the oil rises on the hydrometer scale when the hydrometer in question is immersed in a transparent

oil having a surface tension similar to that of the sample under test.

The temperature of the sample shall be determined from the reading of the separate thermometer placed in the sample, or from that of the thermometer contained in the hydrometer when the thermo-hydrometer type of instrument is used.

NOTE.—Although it is usually satisfactory to determine the temperature of the sample immediately after reading the hydrometer, it is recommended that in all referee tests this temperature be determined both before and after the hydrometer is read.

Method II: For testing fluid bituminous materials, the following method has been standardized:²⁸

The specific gravity of thin fluid bituminous road materials may be determined with the above-mentioned apparatus by first pouring a sufficient quantity of the material into a tin cup, which is then placed in a large dish containing cold or warm water, as occasion may require. The material in the cup should be stirred with the thermometer until it is brought to a temperature of 25° C., after which it should be immediately poured into the hydrometer jar and its gravity determined by means of the proper hydrometer. In case the hydrometer sinks slowly, owing to the viscosity of the material, it should be given sufficient time to come to a definite resting point, and this point should be checked by raising the hydrometer and allowing it to sink a second time. The hydrometer should never be pushed below the point at which it naturally comes to rest until the last reading has been made. It may then be pushed below the reading for a distance of three or four of the small divisions on the scale, whereupon it should immediately begin to rise. If it fails to do so, the material is too viscous for the hydrometer method, and the pycnometer method should be employed.

If the liquid is too opaque for the hydrometer scale to be seen, the reading shall be taken where the meniscus merges into the stem of the hydrometer. The height of the meniscus above the level of the surface shall be estimated in terms of sub-divisions of the scale, and a suitable correction made to arrive at the reading corresponding to the level of the surface of the liquid. For tars and tar products having densities above 1.000 g./ml., hydrometers shall be employed which are adjusted for use in liquids of surface-tension of 35 dynes/cm.

Most hydrometers are adapted to read at 60° F./60° F., or in other words, the instruments are calibrated for water at 60° F. taken as unity. The standard temperature for testing bituminous materials is 77° F., and they should accordingly be brought to this temperature when tested with the hydrometer. For correcting the

reading to water at 77° F., it should be multiplied by 1.002, as follows:

$$\text{Sp. gr. at } 77^{\circ} \text{ F.} / 77^{\circ} \text{ F.} = \text{Sp. gr. at } 77^{\circ} \text{ F. } 60^{\circ} \text{ F.} \times 1.002$$

From the hydrometer reading the following additional corrections may be made:

(a) The difference in density of the liquid between the temperature of the test and the standard temperature.

(b) The difference between the surface tension of the liquid and that for which the hydrometer has been calibrated.

(c) Any scale error of the hydrometer at the point of reading.

The hydrometer has also been used for testing semi-solid bituminous materials,²⁹ as well as for ascertaining the specific gravity of hard asphalts,³⁰ which are melted and tested while fluid at elevated temperatures, the results being converted to 60° F./60° F. by suitable conversion tables.

For converting specific gravity into degrees Baumé and vice versa, the following formulae may be used:³¹

For liquids lighter than water:

$$^{\circ} \text{ Baumé} = \frac{140}{\text{Sp. gr. } 60^{\circ} \text{ F.} / 60^{\circ} \text{ F.}} - 130$$

$$\text{Sp. gr. } 60^{\circ} \text{ F.} / 60^{\circ} \text{ F.} = \frac{140}{130 + ^{\circ} \text{ Baumé}}$$

For liquids heavier than water:

$$^{\circ} \text{ Baumé} = 145 - \frac{145}{\text{Sp. gr. } 60^{\circ} \text{ F.} / 60^{\circ} \text{ F.}}$$

$$\text{Sp. gr. } 60^{\circ} \text{ F.} / 60^{\circ} \text{ F.} = \frac{145}{145 - ^{\circ} \text{ Baumé}}$$

Crude petroleum and refined liquid petroleum products are generally reported in terms of "A.P.I. Gravity"³² as follows:

$$^{\circ} \text{ A.P.I.} = \frac{145.1}{\text{Sp. gr. } 60^{\circ} \text{ F.} / 60^{\circ} \text{ F.}} - 131.5$$

Test 7b. Westphal Balance Method (Used for Thin Fluid Substances). This is well adapted to testing fluid bituminous ma-

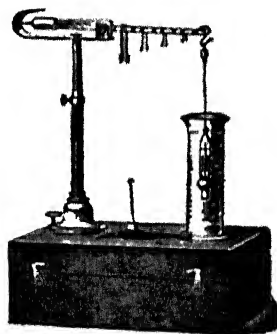
terials. The instrument as supplied by the manufacturer (Fig. 224) is provided with a cylinder of about 50 ml. capacity, calibrated for use at 60° F./60° F. Since the test is generally made at 77° F., it is subject to the same correction as in the hydrometer method.

The Westphal balance may be adapted for as little as 8 ml. of the bituminous material, by using a special plummet, small enough to fit into a 10-ml. cylinder. The plummet may be made from a piece of glass tubing 7 mm. outside diameter, which is sealed at one end with a short platinum wire fused into the glass. Nine to ten grams of mercury are placed in the tube, forming a column 35–40 mm. high. The tube is then cut off within 20 mm. of the top of the mercury column, and the open end sealed with a blowpipe. This plummet should measure 55–60 mm. over all, and weigh from 10 to 12 g. If a represents the weight required to balance the plummet in air, b the weight required to balance the plummet in water at a definite temperature, and c the weight required to balance the plummet in the bituminous material at the same temperature, then the specific gravity of the bituminous material at this temperature may be calculated from the following formula:

$$\text{Specific Gravity} = \frac{c}{b} \cdot \frac{a}{a}$$

By adding a drop of a 1 per cent solution of sodium lauryl-sulfate to the surface of the liquid in the measuring cylinder after immersion of the plummet, the surface-tension effects on the wire will be reduced and more accurate readings obtained.²⁹

Test 7c. Bottle Method (Used for Viscous Fluid and Semi-solid Substances). Several forms of glass bottles are used for this purpose, having a ground-glass stopper with a small vertical hole bored through, to enable it to be filled completely with the bituminous material. These are made in various sizes.



Courtesy of Elmer & Amend.
FIG. 224.—Westphal Balance.

An improvised form, which may be used to good advantage when a small quantity of liquid bituminous material is available, consists of a 1-ml. pipette and a glass tube sealed at one end, the inside diameter of which is slightly larger than the outside diameter of the lower stem of the pipette. On using this instrument, the liquid is first brought to a definite temperature, then sucked to the upper mark of the pipette by means of a piece of rubber tubing temporarily attached to its upper stem. The outside is carefully wiped dry and the lower stem inserted in the glass tube which serves to retain any liquid which may drain from the pipette. A small piece of wire twisted about the pipette near the top is formed into a ring to hang it from the hook above a balance pan. The pipette is thus supported in a vertical position and weighed.²⁴

If a represents the weight of the pipette with glass tube empty, b its weight filled with water at a definite temperature, and c its weight filled with the bituminous material at the same temperature, then the specific gravity may be calculated from the following formula:

$$\text{Specific Gravity} = \frac{c - a}{b - a}$$

If only a small quantity of the substance is available, the following procedure is recommended:²⁵ the specific-gravity bottle is weighed empty (a), a small piece of the substance is pressed against the interior wall of the bottle and again weighed (b). The bottle is thereupon filled with water at 77° F. and again weighed (c). If (d) is the weight of the bottle filled with water at 77° F. alone, then the specific gravity at 77° F. of the substance may be calculated as follows:

$$\text{Specific Gravity} = \frac{b - a}{d + b - (a + c)}$$

Test 7d. Pyknometer Method (Used for Viscous Fluid and Semi-solid Substances).²⁶ This method has been standardized as follows:²⁷

Method 1: The specific gravity of road oils, road tars, asphalt cements and soft tar pitches shall be expressed as the ratio of the weight of a given volume of the material at 25° C. (77° F.) to

that of an equal volume of water at the same temperature and shall be expressed thus:

$$\text{Specific Gravity } 25^{\circ} \text{ C. } (77^{\circ} \text{ F.})/25^{\circ} \text{ C. } (77^{\circ} \text{ F.})$$

The determination of specific gravity shall be made with a pyknometer or weighing bottle (Fig. 225), which shall consist of a straight-walled glass tube approximately 70 mm. long and 22 mm. in diameter, carefully ground to receive an accurately fitting solid glass stopper with a hole of 1.5 to 1.7-mm. bore in place of the usual capillary opening. The lower part of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression shall be about 4.8 mm. at the center. The stoppered tube should have a capacity of about 24 ml. and when empty should weigh not over 35 g.

In lieu of the pyknometer described above, the Hubbard-Carmick type may be used. This type consists of a conical or Erlenmeyer-shaped flask approximately 45 mm. high, 40 mm. in diameter at the bottom, and 25 mm. in diameter at the mouth, carefully ground to receive an accurately fitting solid-glass stopper with a hole of about 1-mm. bore in place of the usual capillary opening. The lower surface of the stopper is made concave in order to allow all air bubbles to escape through the bore. The depth of the cup-shaped depression shall be about 4.8 mm. at the center. The stoppered flask has a capacity of about 25 cc. and when empty weighs about 25 g.

Before making a determination, the pyknometer with stopper shall first be calibrated by weighing it clean and dry upon an analytical balance. This weight is called *a*. It shall then be filled with freshly boiled distilled water at a temperature of 25° C. (77° F.), the stopper firmly inserted, all surplus moisture wiped from the surface with a clean dry cloth and again weighed. This weight is called *b*. When determining the specific gravity of road oils or road tars which flow readily, the material shall be brought to a temperature of 25° C. (77° F.) and poured into the pyknometer until it is full, with care to prevent the inclusion of air bubbles. The stopper is then firmly inserted and all excess of material forced through the opening is carefully removed with a clean dry cloth. The pyknometer and contents are then weighed and this weight is called *c*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c - a}{b - a}$$



FIG. 225.—
Pyknometer
or Weigh-
ing Bottle.

When determining the specific gravity of tar and asphalt products which are too viscous for the method described in the preceding paragraph, a small amount of the material shall be brought to a fluid condition by the gentle application of heat, care being exercised to prevent loss by evaporation. When sufficiently fluid, enough is poured into the clean dry pycnometer to about half fill it. Precautions shall be taken to keep the material from touching the sides of the tube above the final level and to prevent the inclusion of air bubbles. The tube should be slightly warmed before filling. The pycnometer and contents are then cooled to room temperature and weighed with the stopper. This weight is called *c*. The pycnometer is next removed from the balance, filled with freshly boiled distilled water, and the stopper firmly inserted. It is then completely immersed for not less than thirty minutes in a beaker of distilled water maintained at 25° C. (77° F.) after which it is removed, and all surplus water is wiped off with a clean cloth. It is immediately weighed. This weight is called *d*. The specific gravity of the material shall be calculated from the formula:

$$\text{Specific Gravity} = \frac{c - a}{(b - a) - (d - c)}$$

When making the specific gravity determination it is important that:

- (a) Only freshly boiled distilled water shall be used.
- (b) When weighing the pycnometer completely filled, the temperature of its contents shall be within 1° C. (1.8° F.) of 25° C. (77° F.).
- (c) Precautions shall be taken to prevent expansion and overflow of the contents from the heat of the hand when wiping the surface of the pycnometer.
- (d) The presence of all air bubbles shall be eliminated in filling the pycnometer and inserting the stopper.
- (e) Weighings shall be made quickly after filling the pycnometer and shall be accurate to 1 mg. A number of trial fillings and catch weights may be necessary to obtain the desired degree of accuracy.
- (f) To prevent breakage of the pycnometer when cleaning it out after a determination has been made upon a very viscous or semi-solid material, it will be found advisable to warm it in an oven at not over 100° C. until most of the material may be poured out and then to swab it with a piece of soft cloth or cotton waste. When cool it may be finally rinsed with carbon disulfide, benzol or other solvent and wiped clean.

The limit of accuracy of the test is ± 0.005 specific gravity.

Method II: The specific gravity of creosote oil fractions is determined similarly by means of a pycnometer, but in this case the results are expressed at 38° C. (100° F.)/15.5° C. (60° F.). The procedure has been standardized as follows:

Procedure for Fractions Entirely Liquid at 38° C. The specific gravity of creosote fractions (distilled at 235° to 315° C.) entirely liquid at 38° C. shall be determined as follows:

Heat the creosote fraction until completely liquid and continue heating to a temperature not exceeding 38° C.; then pour it into the empty, dry pycnometer until full, avoiding the formation of air bubbles. Insert the stopper in the pycnometer, taking precautions to avoid the inclusion of air bubbles. Place the filled pycnometer in the water bath maintained at a temperature of 38° ± 0.1° C. until the pycnometer and its contents are at a constant volume at 38° C. After immersion in the bath for at least 30 min., remove the pycnometer, wipe clean and dry; then weigh.

Procedure for Fractions Containing Solids at 38° C. The specific gravity of creosote fractions (distilled at 315° to 355° C.) containing solids at 38° C. shall be determined as follows:

Heat the creosote fraction until completely liquid, and pour it into the empty, dry pycnometer until about one-half full, avoiding the inclusion of air bubbles. Cool to room temperature and weigh. Cover the solid or partially solid fraction with freshly boiled distilled water until the pycnometer is about three-quarters full; place in a water bath at 90° C. and allow to remain without agitation until the fraction is liquid and all air is removed. Cool the pycnometer and its contents to a temperature somewhat below 38° C.; fill the pycnometer with freshly boiled distilled water, avoiding the formation of air bubbles; and insert the stopper in the pycnometer, taking precautions to avoid the inclusion of air bubbles.

Place the filled pycnometer in the water bath maintained at a temperature of 38° ± 0.1° C. until the pycnometer and its contents are at a constant volume at 38° C. After immersion in the bath for at least 30 min., remove the pycnometer, wipe clean and dry; then weigh.

The expression "38/15.5° C." means specific gravity of the fraction at 38° C. compared with water at 15.5° C. This cannot be determined directly. The specific gravity is first determined at 38° C. compared with water at 38° C. and this determination represents the relation of the weight of a volume of oil at 38° C. to the weight of an equal volume of water at the same temperature. The relation to an equal volume of water at 15.5° C. is obtained by multiplying the former figure by 0.99393—the density of water at 38° C. compared to water at 15.5° C., (0.99299). From the (0.99905).

foregoing, it will be readily seen that it is incorrect to calculate the specific gravity at 38/15.5° C. by dividing the weight of the oil determined at 38° C. by the weight of water taken at 15.5° C.

Method III: The pycnometer may be used for finding the specific gravity of hard and brittle bituminous substances,³⁸ including hard asphalts of high fusing-point, asphaltites, asphaltic pyrobitumens, non-asphaltic pyrobitumens and pyrobituminous shales. Approximately 3.5 grams of the material ground to 60-mesh are carefully weighed and introduced into a 50-ml. pycnometer, with about 30 ml. of distilled water. A vertical condensing bulb is attached to the pycnometer with a small section of rubber tubing, the open end being connected with an aspirator to maintain a partial vacuum. The pycnometer is then boiled on a water-bath to expel all the air from the sample. The inside of the condensing tube is then washed back into the pycnometer, which is cooled to the desired temperature, stoppered, filled to the mark with water at the same temperature and weighed. The specific gravity may then be calculated from the formula given in (A).

Test 7e. Displacement Method (Used for Semi-solids and Hard Solids). This method has been standardized as follows for finding the specific gravity of semi-solids:³⁹

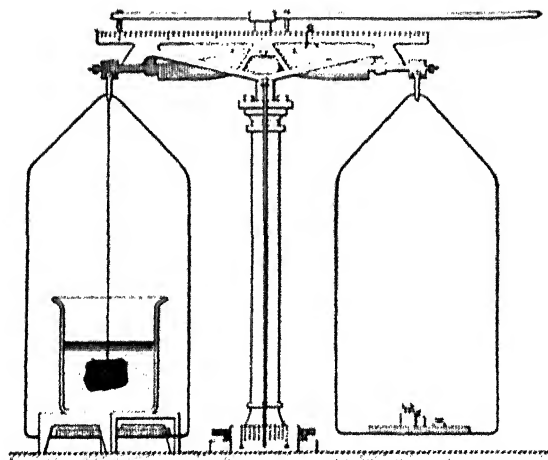
The specific gravity of semisolid bituminous materials may be determined by the displacement method. Weigh a silica crucible suspended from the beam of the balance in air and call the weight *a*, and in water and call the weight *b*. Fill the crucible approximately two-thirds full with the material under examination. Free from bubbles by heating on a hot plate, cool and weigh, calling this weight *c*. Immerse the filled crucible in water at 25° C. for one-half hour, then suspend by a wire from the beam of the balance and weigh it immersed in water at 25° C.; call this weight *d*. The specific gravity is then calculated by means of the following formula:

$$\text{Specific Gravity} = \frac{c - a}{(c - a) - (d - b)}$$

When used for testing hard, solid bituminous materials which can be melted and cast in a mold to form a briquette on cooling, the method is modified as follows:⁴⁰

The determination of specific gravity shall be made with an analytical balance equipped with a pan-straddle or other stationary support (Fig. 226). The test specimen shall be a cube of the

material measuring approximately $\frac{1}{2}$ in. to the edge. It shall be prepared by melting a small sample of the material by the gentle application of heat, care being exercised to prevent loss by evaporation, and pouring when sufficiently fluid into a $\frac{1}{2}$ -in. brass cubical mold, having the form illustrated in Fig. 290, which has been amalgamated with mercury and which is placed on an amalgamated brass plate. Precautions should be taken to prevent the inclusion



Courtesy A. S. T. M.

FIG. 226.—Analytical Balance for Ascertaining the Specific Gravity.

of air bubbles. The hot material should slightly more than fill the mold and when cool the excess may be cut off with a hot spatula. The specimen shall be removed from the mold when cooled to room temperature.

The balance shall first be tared with a piece of fine waxed silk thread sufficiently long to reach from the hook on one of the pan supports to the straddle or rest. The test specimen shall then be attached to the thread, so as to be suspended about 1 in. above the straddle from the hook on the pan support, and weighed. This weight is called *a* and shall be accurate to 0.1 mg. The specimen, still suspended by the thread, shall then be weighed completely immersed in freshly boiled distilled water at 25° C. (77° F.) $\pm 1^\circ$ C. (1.8° F.), adhering air bubbles being first removed with a fine wire. This weight is called *b* and shall be accurate to 0.1 mg. The specific gravity of the material shall be calculated from the formula :

$$\text{Specific Gravity} = \frac{a}{a - b}$$

The limit of accuracy of the test is ± 0.005 specific gravity.

VOIDS

Test 7f. Voids (Entrapped Air). The voids in asphalts, asphalt-filler mixtures and bituminized fabrics may be rapidly deter-

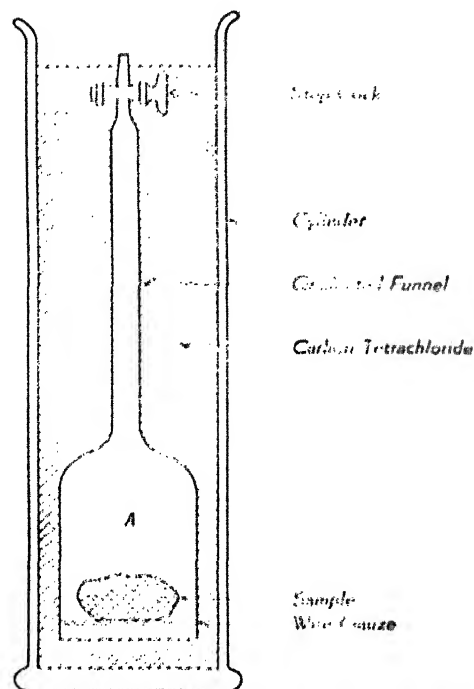


FIG. 227.—Apparatus for Ascertaining Voids (Entrapped Air).

mined as follows: The volume of the specimen is first ascertained from its specific gravity, or in the case of bituminized fabrics, from its linear dimensions and thickness in cms. The sample weighing 25 to 50 g. is then placed in the reservoir "A" of a graduated funnel, as shown in Fig. 227. A copper wire-gauze is inserted in the

bottom to support the sample, whereupon the funnel is inverted, and with the stop-cock open, immersed in a cylinder filled with carbon tetrachloride. When the funnel is full and the carbon tetrachloride passes through the stop-cock, the latter is quickly closed. The cylinder containing the funnel is placed in a water-bath maintained at 50° C. The entrapped air rises to the top and displaces the carbon tetrachloride. After the bubbling has ceased, the cylinder and contents are cooled to 20° C., and the volume of air is then read. This divided by the volume of the sample gives the volume percentage of the voids. When tested in this manner, residual asphalt showed 2 per cent of air; filled asphalt 6 per cent; asphalt roll-roofings 2 to 18 per cent by volume of entrapped air (i.e., voids).

COLLOIDAL CAPACITY

The tests included under this heading are designed for the purpose of measuring the capacity of bituminous substances to maintain colloidal dispersions of finely divided inorganic materials. This depends upon the characteristics of the bituminous substance, as well as the nature and physical subdivision of the inorganic material present. Colloidal dispersions of this type are characterized by the fact that the inorganic material remains in more or less permanent suspension upon dissolving the bituminous substance in carbon disulfide or benzol. The test is of value in measuring the stability of bituminous substances combined with finely divided inorganic materials and the ability of the latter to remain in dispersion. The following tests have been proposed for the purpose:

Test 7g. Clay Dispersions. This test has been devised by Clifford Richardson,⁴¹ and measures the capacity of the bituminous substance to maintain a dispersion of colloidal clay. A suitable type of colloidal clay is first selected and dispersed in water. This dispersion is then introduced into the bituminous substance maintained in a liquid condition below the temperature of boiling water. Upon removing the aqueous phase from the emulsion by heating, the highly dispersed clay will be associated with the bituminous phase, and become dispersed in a colloidal state to an extent depending upon the colloidal capacity of the bituminous substance. Tests were made by the foregoing method to introduce colloidal clay into a number of solid residual asphalts derived from various

types of petroleum, which resulted in a product of approximately 33½ per cent clay combined with 66½ per cent asphalt, after removal of the water. The results given in Table CXVIII were recorded:

TABLE CXVIII
DISPERSION OF COLLOIDAL CLAY IN ASPHALT

	Original Penetration at 77° F. (Test 96)	Quantity of Colloidal Clay in Dispersion	Mixture Maintained in Tubes 24 Hrs. at 325° F.		Clay Remaining in Suspension Upon Centrifuging a 10% Solution in Carbon Disulfide
			Clay Remaining in Suspension	Hence Degree of Sedimentation	
		Per Cent	Per Cent	Per Cent	Per Cent
Trinidad residual asphalt. . .	50	33.5	33.7	0.0	2.23
Venezuela residual asphalt*. .	48	32.4	30.1	7.0	1.91
Mexican residual asphalt. . .	50	33.3	27.2	18.3	1.89
California residual asphalt. .	50	31.8	23.8	25.2	0.94
Mid-continental semi-asphaltic residual asphalt. . .	51	33.8	21.7	35.8	0.54

* Derived from Bababui heavy petroleum

Test 7h. Ultramicroscopic Count of Colloidal Particles. The ultramicroscope consists essentially of a compound microscope arranged for examining in a dark field an intense convergent beam of light cast upon the particles of the substance under examination, so that the latter will diffuse the light as viewed by the eye. An intense, but minute beam of light is focused on the fluid contained on a glass slide or in a small cell, above which a compound microscope is adjusted vertically, so that the narrowest part of the light cone occupies the center of the focal field. If the fluid under examination is optically clear, or if it contains particles so minute that they cannot diffract sufficient light to create a visual impression, the light cone cannot be seen. If, however, enough light is diffracted by the particles, they become visible against a dark field. Particles may thus be observed that are beyond the resolving power of an ordinary microscope.

The following table will indicate the relative sizes of particles examined by various methods:

Material Unit	Diameter of Particles	Method of Examination
Atoms.....	0.1-2.5 $\mu\mu$	X-ray spectrometer
Molecules.....	0.5-5 $\mu\mu$	" "
Molecular groups.....	1-10 $\mu\mu$	Ultramicroscope
Primary colloidal particles.....	2-20 $\mu\mu$	"
Secondary colloidal particles.....	5-100 $\mu\mu$	"
Microscopically resolved particles.....	Over 250 $\mu\mu$	Ordinary microscope
Particles visible to the eye.....	About 10 μ	Unaided eye.

Note: $1\mu = 0.001$ mm. = 1 micron.

$1\mu\mu = 0.001\mu = 0.00001$ mm.

Particles retained by filter paper measure above 1μ ; particles which pass through filter paper measure up to $100\mu\mu$; particles beyond the resolving power of the ordinary microscope, but which are visible under the ultra-microscope ($250\mu\mu$) are termed "ultra-microns" or "sub-microns." If they cannot be discerned with the ultra-microscope, they are termed "amicros." Rapid Brownian movement is observed when particles measure less than $10\mu\mu$ in diameter; slow Brownian movement occurs with particles which measure between $10\mu\mu$ and 1μ ; and no Brownian movement occurs when the particles measure greater than 1μ .

The best solvents for use in dissolving bituminous substances in their ultramicroscopic examination are carbon disulfide and benzol, since these have about the same surface-tension as the substance itself, in the case of asphalts. Nitrobenzol is recommended for tars and pitches. The ultramicroscopic image of asphalts is not easy to obtain, since the ultramicros are very minute and not very luminous. A powerful source of light and painstaking focusing are required. Natural asphalts show bright particles of mineral matter which are coarser than the ultramicros of carbon present in petroleum asphalts. An 0.1 to 0.01 per cent solution of coal tar or coal-tar pitch in nitrobenzol gives clear images, showing many particles in active Brownian movement. Such particles are not as luminous as those of natural asphalt, or mixtures of petroleum asphalt with fine inorganic fillers. Dark-field illuminators are fitted to the sub-stage of the microscope in place of the usual Abbé sub-stage condenser, and must be precisely centered with respect to the

optical axis of the microscope. The usual arrangement is shown in Fig. 228.

The light beam should be introduced through an aperture of 1.00 to 1.40. The object should be covered with a thin cover-glass, which serves the purpose of totally reflecting the light beam.



Courtesy of Carl Zeiss, Inc.

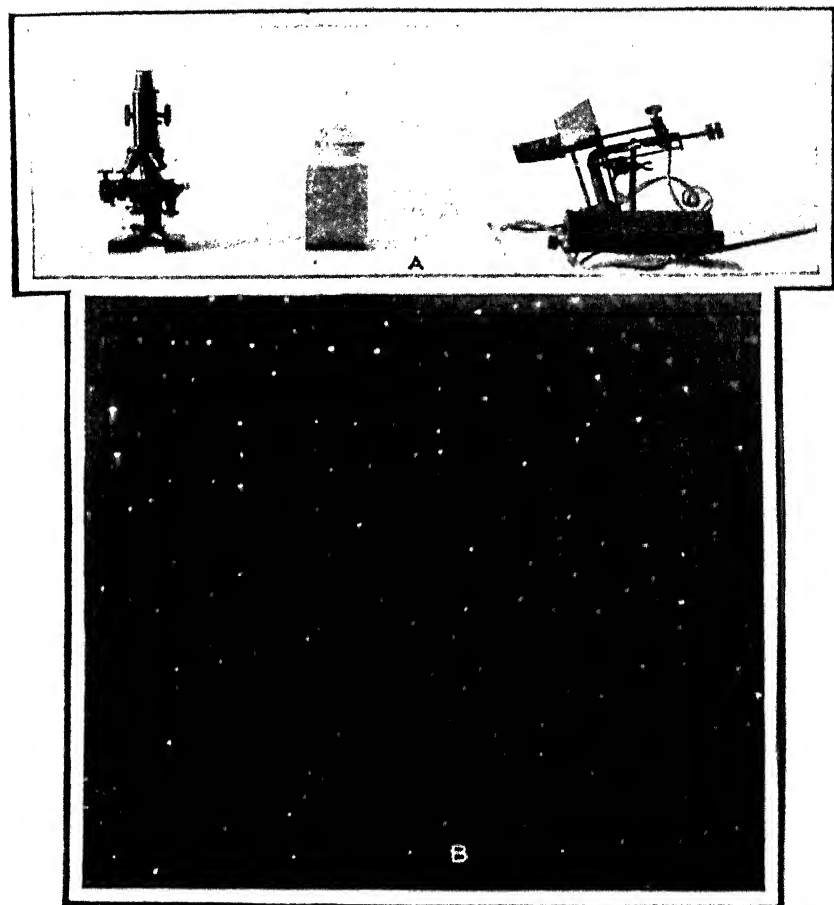
FIG. 228.—Ultramicroscope with Dark-field Illuminator. A—High Powered Binocular Microscope; B—Sub-stage Dark-field Condenser; C—Source of Illumination.

Oil or water immersion should be employed for the objective, which should have a minimum aperture of less than 0.8.⁴²

The following method involves counting the colloidal particles in a predetermined volume of a benzol solution of the bituminous substance at a dilution of 1 to 5000. The test is made as follows:⁴³

Description of the Microscope. Use an ordinary microscope capable of giving a linear magnification of from 50 to 740 diameters at a tube-length of 160 mm. Use an eyepiece "×7.5" and an objective "4 mm.," magnifying 320 diameters. The microscope

should be equipped with a mechanical stage for lateral orientation as well as an accurately calibrated micrometer screw for recording



Courtesy E. C. F. Ford

FIG. 229.—Microscope for Counting Colloidal Particles. A—Assembly of Apparatus. B—Appearance of Field Showing Colloidal Particles.

vertical measurements. The eyepiece should carry a counting device consisting of a cross-line micrometer scale with a ground glass border divided into 25 squares, each side of which measures 1.25

mm. and corresponds to exactly 0.05 mm. of a stage micrometer at a tube-length of 160 mm. A ray of light from an arc lamp is passed through a cooling bath, acting as a ray filter, composed of 10 mgm. diamine green dissolved per liter of distilled water, and by means of a mirror at the base of the microscope projected through a substage paraboloid condenser having a central stop, which serves to bring the rays to a sharp focus. In this manner, the colloidal particles, whose indices of refraction vary from that of the enclosing liquid, become luminously visible, whereas the re-

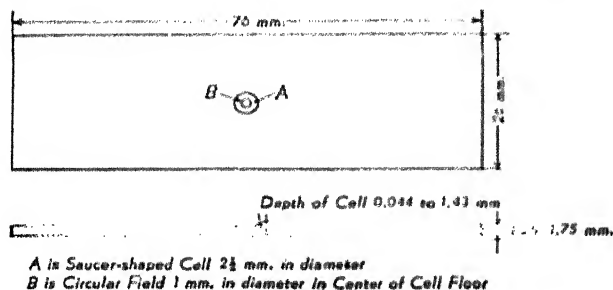


FIG. 230.—Cell for Counting Colloidal Particles.

mainder of the field remains perfectly dark. The microscope is set up as illustrated in Fig. 229.

Description of the Cell. The cell, illustrated in Fig. 230, consists of a 26×76 mm. object glass between 1.25 and 1.75 mm. thick, having a cavity excavated therein $2\frac{1}{2}$ mm. in diameter and not exceeding 0.10 mm. deep with a corresponding capacity of less than 0.10 cmm. A circle 1 mm. in diameter is inscribed at the bottom of the cell in its center with a diamond-point marker. The cell and cover glass must be cleaned preparatory to filling, by boiling in concentrated sulfuric acid, rinsing in water, alcohol and benzol, drying with soft cotton cloth and rubbing with optical tissue paper.

Preparation of the Solutions. To prevent Brownian movement of the particles, they are examined in a viscous solution of colloid-free asphaltic residual oil containing paraffin, prepared by fluxing 2.5 g. Mexican petroleum asphalt (penetration 148 at 77° F.) with 0.5 g. crystalline paraffin, and then diluting to 100 ml. with benzol

containing 10 per cent ethyl alcohol. This is evaporated to constant weight on a water-bath, diluted to its original volume with benzol and passed several times through an alundum filter tube packed with macerated filter paper until not more than 10 particles are visible per $\frac{1}{4}$ mm. square, when examined in accordance with the method about to be described. Dissolve 1 g. of the specimen to be examined in 50 ml. benzol in a stoppered centrifugal tube; let stand overnight and then centrifuge for 1 hour at a speed of 800 revolutions per minute. With pipette draw off 1 c. mm. of the liquid at a depth of 10 mm., transfer to a glass-stoppered graduate, and dilute to 10 ml. with the paraffin-asphaltic oil dilutant. By this means a dilution of 1 to 5000 of the colloids present in the original sample is obtained.

Counting the Colloidal Particles. With a pipette, rapidly transfer 1 or 2 drops of the properly diluted solution to a cleaned cell and cover immediately with an 18-mm. cover glass, pressing it with a bluntly pointed wooden rod to expel the excess liquid and secure close contact. When the solution exuding from the cover glass hardens by evaporation, seal the cover glass with a 30 per cent solution of Canada balsam in ether. Transfer the slide to the microscope stage, and place upon it a few drops of an immersion liquid, such as cedar oil or glycerin, to prevent loss of light through refraction of the rays issuing from the condenser. In correct focus, the colloidal particles will appear as brilliant points of light against a dark background as illustrated in Fig. 229 (B). They should be evenly distributed and in constant, though restricted, motion. Examine the area within the central millimeter circle, representing exactly $\frac{1}{4}$ sq. mm., equivalent to four fields of the cross-line eyepiece-micrometer. Count the number of particles in each of the 25 square subdivisions of the micrometer, throughout the entire depth of liquid. From the average of four such determinations, compute the number of colloidal particles per c. mm. of solution at 1:5000 dilution.

Results Recorded. Table CXIX contains a summary of the observations made on some typical bituminous substances.

The copper carbonate salts become largely reduced to red cuprous oxide, accompanied by an enormous colloidal dispersion, while with the remaining salts the reduction is much less complete

(*i* and *j*) or entirely lacking (*k* and *l*) and the development of colloids correspondingly less. The accuracy of the method depends chiefly upon the care exercised in the construction of the cell, as well as upon the proper consistency and optical purity of the diluting liquid.

TABLE CXIX
COLLOIDAL PARTICLES IN TYPICAL ASPHALTS

	Penetration at 77° F. (Test 56)		Colloidal Particles	
	Before Treat- ment	After Treat- ment	Average per cmm. at 1 to 3000 Dilution	Relative Standing
(a) Refined Trinidad asphalt	2		406,256	100.0
(b) Refined Trinidad asphalt combined with 3.3% colloidal clay (Note 1)	2		30,539	7.5
Residual asphalt from Trinidad petroleum:				
(c) Combined with 3.3% clay (Note 2)		235	169,262	41.6
(d) Heated to 340° F. with 16½% CuCO ₃ ·Cu(OH) ₂		28	1,685,950	367.5
(e) Heated to 340° F. with 10% CuCO ₃ ·Cu(OH) ₂		25	285,500	101.5
Residual asphalt from Mexican petroleum:				
(f) Combined with 3.3% clay (Note 2)	135	13	408,114	26.6
(g) Heated to 340° F. with 16½% CuCO ₃ ·Cu(OH) ₂	57	8	4,858,000	293.5
(h) Heated to 340° F. with 10% CuCO ₃ ·Cu(OH) ₂	57	10	1,501,800	249.5
(i) Heated to 340° F. with 26% PbO ₂ ·H ₂ O	250	70	362,300	64.6
(j) Heated to 340° F. with 10% CuSO ₄	57	16	331,100	50.3
(k) Heated to 340° F. with 10% FeSO ₄	57	28	18,667	4.6
(l) Heated to 340° F. with 10% ZnSO ₄	57	16	4,159	1.1
(m) Gilsonite heated to 340° F. with 10% CuCO ₃ ·Cu(OH) ₂	0	0	100,711	24.8
(n) Water-gas tar heated to 340° F. with 20% CuCO ₃ ·Cu(OH) ₂		21	20,455	5.0

NOTE 1.—All insoluble organic and mineral matter removed before mixing with the clay (see Note 2), by dissolving in benzol, adding 2 per cent shellac dissolved in alcohol, evaporating to constant weight, redissolving in benzol, and finally filtering through an abundance tube, so the solution is essentially free from colloidal particles.

NOTE 2.—Incorporated by Clifford Richardson's method of heating an aqueous emulsion of clay and asphalt, until all the moisture is expelled. See Test 72-1.

The following method has been proposed for examining road tars:⁴⁴ Dissolve 0.2 to 0.3 g. of the tar in C.P. nitrobenzol (with the least possible agitation), so as to form exactly 1 volume per cent solution (having first ascertained the specific gravity of the substance). The solution is warmed on a water-bath to 25° C.; cooled to room temperature; then filtered through a 7-cm. Schleicher & Schüll No. 595 filter to remove the coarse particles. One drop of the well-shaken filtrate is transferred to an accurately scaled slide-glass (divided into squares 0.05 mm.—such as a Thoma hemocytometer) in a layer 0.1 mm. deep, and examined under an ultra-microscope under a magnification of $\times 400$. After allowing the

slide to stand 1-2 minutes, the "micella" are counted in various squares and the average computed. In making the count, any aggregation of particles are taken as a single particle. The average multiplied by 400,000 gives the number of micella per cubic millimeter of tar.

It is contended that the greater the number of micella, the better will be the binding power of the tar. In Holland (where this test originated), specifications call for a minimum of 10 million micella for good road tars. The number of micella is influenced by the age of the specimen, the temperature to which it has been heated, and the extent of mechanical manipulation (i.e., stirring or agitation). The foregoing test must therefore be performed under carefully controlled conditions.⁴⁵

(B) MECHANICAL TESTS

VISCOSITY

This test is of value in determining the adaptability of the bituminous substance for a given purpose, for gauging the uniformity of supply, and for factory control work. It is used particularly for examining liquid to semi-liquid substances for road purposes, and may also be used to good advantage for predetermining the ability of semi-solid substances to saturate fabrics at elevated temperatures.

It has been observed that the viscosity of mixtures is invariably lower than an additive result of computations.⁴⁶ With most bituminous substances, the relation between viscosity and temperature may be expressed by a simple exponential function:

$$v \times t^n$$

where v = viscosity; t = temperature; and " n " also " a " represent two constants. If the logarithm of viscosity is plotted against the logarithm of temperature, a straight-line relationship will result.⁴⁷

The addition of equal bulk volumes of different fillers to the same quantity of bituminous binder is claimed to give products of the same viscosity, irrespective of the nature of the filler.⁴⁸

It has also been proposed to determine the viscosity of tars and pitches in terms of the so-called "equi-viscous temperature," which

represents the particular temperature at which the material will exhibit a predetermined viscosity.⁴⁹ For example, in using the B.R.T.A. viscosimeter as standardized by the Standardization of Tar Products Tests Committee of Great Britain⁵⁰ this temperature is one at which the tar attains a viscosity of exactly 50 seconds. Another consists in finding the viscosity of the material at two or more temperatures, in a modified form of Redwood viscosimeter (provided with a 10-mm. orifice) then plotting the results logarithmically, and by interpolation, ascertaining the temperature at which the line crosses the "log. 50-sec." line. It has been reported that the range of commercial road tars is from 20° to 55° C., and for briquetting pitch in the neighborhood of 100° C. equi-viscous temperature. It has also been noted that the equi-viscous temperature of a mixture of the two tars corresponds closely to the mean of the equi-viscous temperature of the components; furthermore, that the reduction in equi-viscous temperature of a tar caused by the addition of flux oil, is approximately proportional to the amount of oil added.

Standard tables have also been formulated showing the viscosity-temperature relationship of petroleum products.⁵¹

Test 8a. Engler Method. The Engler method of test has been standardized as follows:⁵²

Apparatus. The viscosity of fluid bituminous road materials may be determined at any suitable temperature by means of the Engler viscosimeter. This apparatus is shown in Fig. 231, and may be described as follows: *a* is a brass vessel for holding the material to be tested, and may be closed by the cover *b*. To the conical bottom of *a* is fitted a conical outflow tube *c*, exactly 20 mm. long, with a diameter at the top of 2.9 mm. and at the bottom of 2.8 mm. This tube can be closed and opened by the pointed hardwood stopper *d*. Pointed metal projections are placed on the inside of *a* at equal distances from the bottom, and serve for measuring the charge of material, which is 240 cc. The thermometer *e* is used to ascertain the temperature of the material to be tested.

The vessel *a* is surrounded by a brass jacket *f*, which holds the material used as a heating bath, either water or cottonseed oil, according to the temperature at which the test is to be made. A tripod *g* serves as a support for the apparatus and also carries a ring-burner *h*, by means of which the bath is directly heated. The meas-

using cylinder of 50-cc. capacity, which is sufficiently accurate for work with road materials, is placed directly under the outflow tube.

Calibration. As all viscosity determinations shall be compared with water at 25° C., the apparatus shall be calibrated as follows: The cup and outlet tube shall first be scrupulously cleaned, using ether to remove all traces of oil, then followed with an alcohol wash and thoroughly dried. A piece of soft tissue paper is convenient for cleaning the outlet tube. The stopper shall then be inserted in the tube and the cup filled with water at 25° C. to the top of the projections. The measuring cylinder shall be placed directly under the outflow tube, so that the effluent will not touch the sides. The stopper shall then be completely withdrawn from apparatus. The stopper hole in the viscosimeter shall be open during the efflux of the material. The time required for 50 ml. to flow out shall be ascertained by means of a stop watch, and the results so obtained should be checked a number of times. The time required for 50 ml. of water should be about 11 sec.

Procedure. Bituminous road materials are tested in the same manner as water, and the temperature at which the test is made is controlled by the bath. The material should be brought to the desired temperature and maintained there for at least 3 min. before making the test. The results are expressed as specific viscosity compared with water at 25° C., as follows:

$$\text{Specific viscosity} \left. \begin{array}{l} \text{at } t^{\circ} \text{ C.} \end{array} \right\} = \frac{\text{Seconds for passage of 50 cc. at } t^{\circ} \text{ C.}}{\text{Seconds for passage of 50 cc. of water at } 25^{\circ} \text{ C.}}$$

The bituminous material is ordinarily tested at 77° F. (25° C.), 172° F. (50° C.), or 212° F. (100° C.) depending upon its consistency. The viscosimeter is filled to the top of the points with bituminous material brought to the required temperature, and the time noted for 200, 100, 50 or 20 ml. to flow through the orifice. If 100 ml. are allowed to flow through the instrument, the reading should be multiplied by 2.35 to calculate the time of flow for 200 ml. If 50 ml. are allowed to flow through, the reading should be

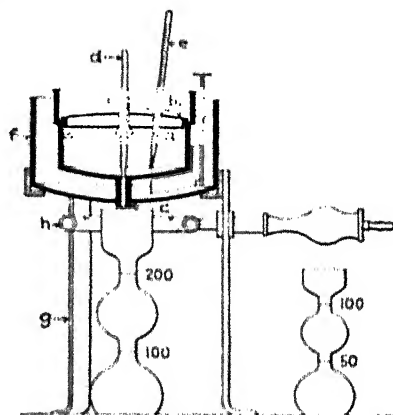


FIG. 231.—Engler Viscosimeter.

multiplied by 5, and with 20 ml. by 11.95 to obtain the time of flow for 200 ml.

If insufficient material is available to fill the viscosimeter, either 25 or 45 ml. may be introduced, and 10 or 20 ml. respectively allowed to flow through, in which events the readings should be multiplied by 13 or 7.25 respectively to obtain the time of flow for 200 ml. These factors are constant. The viscosity recorded at t° F. compared with water at 68° F. is equal to the number of seconds for 200 ml. of the substance to pass through at t° F. divided by the seconds for 200 ml. of water to pass through at 68° F. Tables have been worked out showing the factor to be used when the apparatus is filled with smaller volumes of liquid allowing different amounts to flow through.⁵³ For testing roofing saturants, the time of flow of 50 ml. at 350° F. should be recorded.

Test 8b. Saybolt Method. This test has been standardized as follows: ⁵⁴

The Saybolt Universal Viscosimeter shall be used only for substances with times of flow of more than 32 sec. There is no maximum limit to viscosity to be measured by the Saybolt Universal Viscosimeter but, in general, liquids having an outflow of the order of 1000 sec. and higher, Saybolt Universal, are tested more conveniently by means of the Saybolt Furol Viscosimeter.

The Saybolt Furol Viscosimeter shall be used only for substances with times of flow of more than 25 sec. The outflow time of the Furol (Note) instrument is approximately one-tenth that of the Universal.

NOTE.—The word "Furol" is a contraction of the phrase "fuel and road oils."

The apparatus shall consist of an oil tube, bath, receiver, thermometers, timer, and withdrawal tube, conforming to the requirements specified in the following Paragraphs (a) to (g):

(a) *Oil Tube.* The oil tube (illustrated in Fig. 232A) shall be entirely of corrosion-resistant metal, with or without plating, and shall conform to the dimensional requirements shown in Table CXX within the permissible variations prescribed. The lower end of the oil tube shall be provided with a nut for locking it in place in the bath and with a cork or other suitable device to prevent flow until the test is started. For convenience, a string or its equivalent may be attached to the cork for rapid removal.

The oil tube shall be standardized, and any correction in excess of 0.2 per cent shall be applied. The time of flow shall be within 1 per cent of the time as obtained with the National Bureau of Standards' master Saybolt oil tube.

(b) *Bath.* The bath shall serve as a support to hold the oil-tube in a vertical position and as a container for the bath liquid. The bath shall be equipped with a stirring device and with means for heating or cooling. The source of heat or refrigeration shall

TABLE CXX
DIMENSIONS OF OIL TUBES

Dimensions	Saybolt Universal Viscosimeter			Saybolt Furoi Viscosimeter		
	Minimum, cm.	Normal, cm.	Maximum, cm.	Minimum, cm.	Normal, cm.	Maximum, cm.
Inside diameter of outlet tube	0.1750	0.1755	0.1760	0.115	0.115	0.117
Outside diameter of outlet tube at lower end	0.28	0.30	0.32	0.40	0.41	0.46
Length of outlet tube*	1.215	1.215	1.215	1.215	1.215	1.215
Height of overflow rim above bottom of outlet tube*	12.40	12.50	12.60	12.40	12.50	12.60
Outside diameter of overflow rim, at the top*†	†	†	3.30	†	†	3.30
Diameter of container*	2.953	2.975	2.995	2.955	2.975	2.995
Depth of cylindrical part of container*	8.8			8.8		
Diameter of container between bottom of cylindrical part of container and top of outlet tube*	0.9			0.9		

* This dimension is identical in the Saybolt Universal and the Saybolt Furoi instruments.

† The minimum value shall preferably be not less than 3.2 cm.

‡ The section of overflow rim shall be bounded by straight lines, except that a fillet is permissible at the junction with the bottom of the gallery.

be more than 1¼ in. (3 cm.) from the oil tube. If an external heater is used, it must be more than 2 in. (5 cm.) from the oil tube. The bath temperature necessary to maintain thermal equilibrium (while the oil in the tube is well stirred by the oil-tube thermometer) shall be within ±0.1° F. (0.06° C.) of the standard temperatures of 70° F. (21.1° C.) or 77° F. (25° C.), or shall not exceed 100.25°, 122.35°, 130.5°, 141.0°, 181.5° or 212.0° F. (37.9°, 50.2°, 54.7°, 60.6°, 83.1° or 100.0° C.), respectively, for the standard temperatures mentioned below. The level of the bath liquid shall not be lower than ¼ in. (0.5 cm.) above the overflow rim of the oil tube. For referee testing, the bath liquid must be one which in the bath used will meet the preceding bath-temperature conditions (see Note).

NOTE.—These temperature requirements can be met with water, aqueous solutions, and some baths with oil. In routine testing oil is generally used as the bath medium. This is allowable provided the temperature of the oil bath is adjusted so that the neces-

sary condition of thermal equilibrium is maintained. It may be necessary to maintain the oil bath at slightly higher temperatures than those specified in the preceding paragraph. Temperature differentials between oil bath and oil tube necessary to maintain thermal equilibrium may be double those given above.

(c) *Receiver.* The receiving flask (see Fig. 232B) shall be of glass with a capacity up to the graduation mark on its neck of 60 ml. \pm 0.05 ml. at 68° F. (20° C.). At the graduation mark, the

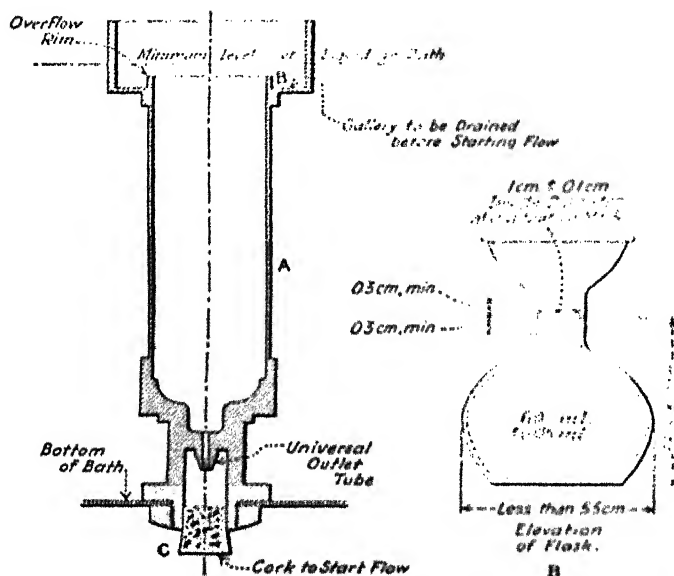


FIG. 232.—Saybolt Viscosimeter.

Courtesy A.S.T.M.

inside diameter of the neck of the flask shall be 1 cm. \pm 0.1 cm. The cylindrical portion of the neck of the flask shall extend not less than 0.3 cm. above and below the graduation mark. The graduation mark shall be 5.8 cm. \pm 1.0 cm. above the bottom of the flask. The maximum outside diameter shall be less than 5.5 cm.

(d) *Oil-tube Thermometers.* The oil-tube thermometers shall conform to the following requirements. They shall include two sets of six thermometers each, one set being graduated in Fahrenheit degrees and the other in Centigrade degrees, the ranges being chosen to include the temperatures commonly used in testing. To prevent contact of the thermometer with the orifice in the oil tube

a suitable support shall be attached to the enlargement of the thermometer stem.

(c) *Bath Thermometers.* Oil-tube thermometers, or other means for measuring temperature of at least equal accuracy, shall be used in the bath.

(f) *Timer.* The stop watch or other timing device used shall be graduated in divisions of 0.2 sec. or less, and shall be accurate to within 0.1 per cent when tested over a 60-min. period (see Note).

NOTE.—Electrical timing devices are permissible provided they are accurate and capable of being read to 0.2 sec. Timing devices actuated by synchronous motors shall be used only on electric circuits of controlled frequency.

(g) *Withdrawal Tube.* The tube or pipette used for draining the gallery shall have a smooth tip of about 3 mm. outside diameter and about 2 mm. inside diameter.

With the Saybolt Universal Viscosimeter, determinations shall be made at 77°, 100°, 130° or 210° F. (21.1°, 37.8°, 54.4° or 98.9° C.).

With the Saybolt Furol Viscosimeter, determinations shall be made at 77°, 100°, 122° or 210° F. (25°, 37.8°, 50° or 98.9° C.). In tests on road and paving materials, determinations may also be made at 140° and 180° F. (60° and 82.2° C.).

Viscosity determinations shall be made in a room free from drafts and rapid changes in temperature. For standardization and referee tests the room shall be between 68° and 86° F. (20° and 30° C.) and the actual temperature shall be recorded. For routine tests, temperatures up to 100° F. may be employed without introducing errors in excess of 1 per cent. Determinations shall not be made at temperatures below the dew point of the atmosphere surrounding the instrument. The oil tube shall first be cleaned with an effective solvent, such as benzol, and excess solvent shall be removed from the gallery.

All oil shall be passed through a 100-mesh wire strainer before it is introduced into the oil tube. After the tube is cleaned, a quantity of the oil to be tested, sufficient to wet the entire surface of the tube, shall be poured into the tube and allowed to drain out (see Note). The cork stopper shall be inserted not less than $\frac{1}{4}$ in. nor more than $\frac{3}{8}$ in. into the lower end of the air chamber at the bottom of the oil tube. The cork shall fit tightly enough to prevent

the escape of air, as evidenced by the absence of oil on the cork after it is withdrawn.

NOTE.—The plunger commonly supplied with the viscosimeter shall never be used on instruments maintained as standards.

If the test temperature is above that of the room, the oil shall be heated to not more than 3° F. (1.7° C.) above the temperature of test and if the temperature is below that of the room, the oil shall be cooled to not more than 3° F. (1.7° C.) below the temperature of test. In no case, however, shall the oil be preheated to a temperature above 50° F. (27.8° C.) below the flash point. The oil shall be poured into the oil tube until it ceases to overflow into the gallery. The oil in the oil tube shall be kept well stirred with the oil-tube thermometer, care being taken to avoid hitting the outflow tube. The bath temperature shall be adjusted until the oil temperature remains constant. After thermal equilibrium has been attained, no further adjustments shall be made in the bath temperature. The test result shall be discarded if the indicated bath temperature varies by more than $\pm 0.05^{\circ}$ F. (0.03° C.) in tests at 70° , 77° , 100° , 122° and 130° F. (21.1° , 25° , 37.8° , 50° and 54.4° C.), or by more than $\pm 0.10^{\circ}$ F. (0.06° C.) in tests at 140° , 180° and 210° F. (60° , 82.2° and 98.9° C.).

After the temperature of the oil in the oil tube has remained constant within 0.02° F. (0.01° C.) of the desired temperature for 1 min. with constant stirring, the oil-tube thermometer shall be withdrawn and the surplus oil removed quickly from the gallery by means of the withdrawal tube, so that the level of the oil in the gallery is below the level of the oil tube proper. The tip of the withdrawal tube shall be inserted at one point in the gallery. The test shall be started over again if the tip of the withdrawal tube touches the overflow rim. Under no conditions shall the excess oil be removed by rotating the withdrawal tube around the gallery.

The receiving flask shall be placed in position, so that the stream of oil from the outlet tube will strike the neck of the flask. The graduation mark on the receiving flask shall be not less than 10 cm. or more than 13 cm. from the bottom of the bath. The cork shall be snapped from its position and at the same instant the timer shall be started. The timer shall be stopped when the bottom of the

meniscus of the oil reaches the mark on the neck of the receiving flask.

The time in seconds as determined by the prescribed procedure, with the proper calibration correction, is the Saybolt Universal (or Saybolt Furol) viscosity of the oil at the temperature at which the test is made. Results shall be reported to the nearest 0.1 sec. for viscosity values below 200 sec. and to the nearest whole second for values 200 sec. or above. With proper attention to details of procedure, results in different laboratories with different operators under referee or standardization conditions of testing, should not differ by more than 0.5 per cent.

It has been reported that the Saybolt-Furol viscosity at any particular temperature is approximately 4 times the Engler specific viscosity at the same temperature.

Test 8c. Absolute Viscosity. The "poise" is the unit used to measure the absolute viscosity of fluids. When a fluid fills the space between two planes 1 cm. apart, each measuring 1 sq. cm., the tangential force in dynes which must be exerted to move one of the planes parallel to the other, exactly 1 cm. in 1 sec., represents the absolute viscosity of the fluid in poises (μ).

Method 1: The absolute viscosity of bituminous substances may be ascertained by means of a graduated capillary tube to which suction is applied.⁶⁸

Fig. 233A shows the viscosimeter proper in detail, and Fig. 233B the schematic arrangement of the apparatus when assembled. Approximately 5 cm. of the substance are placed in the sample-cup, the upper level of which should fall ± 2 mm. from the 10 mm. graduation on the capillary tube, representing a depth of immersion approximately 1.0 cm. When the viscosimeter and contents have been brought to the desired temperature, the vacuum reservoir is evacuated to an extent (measured in cms. of mercury) sufficient to cause the material under test to rise in the capillary at a speed neither too rapid nor unnecessarily slow. Open the stop-cock in the line, and with a stop watch determine the number of seconds required for the interface to pass between two predetermined marks on the graduated capillary.

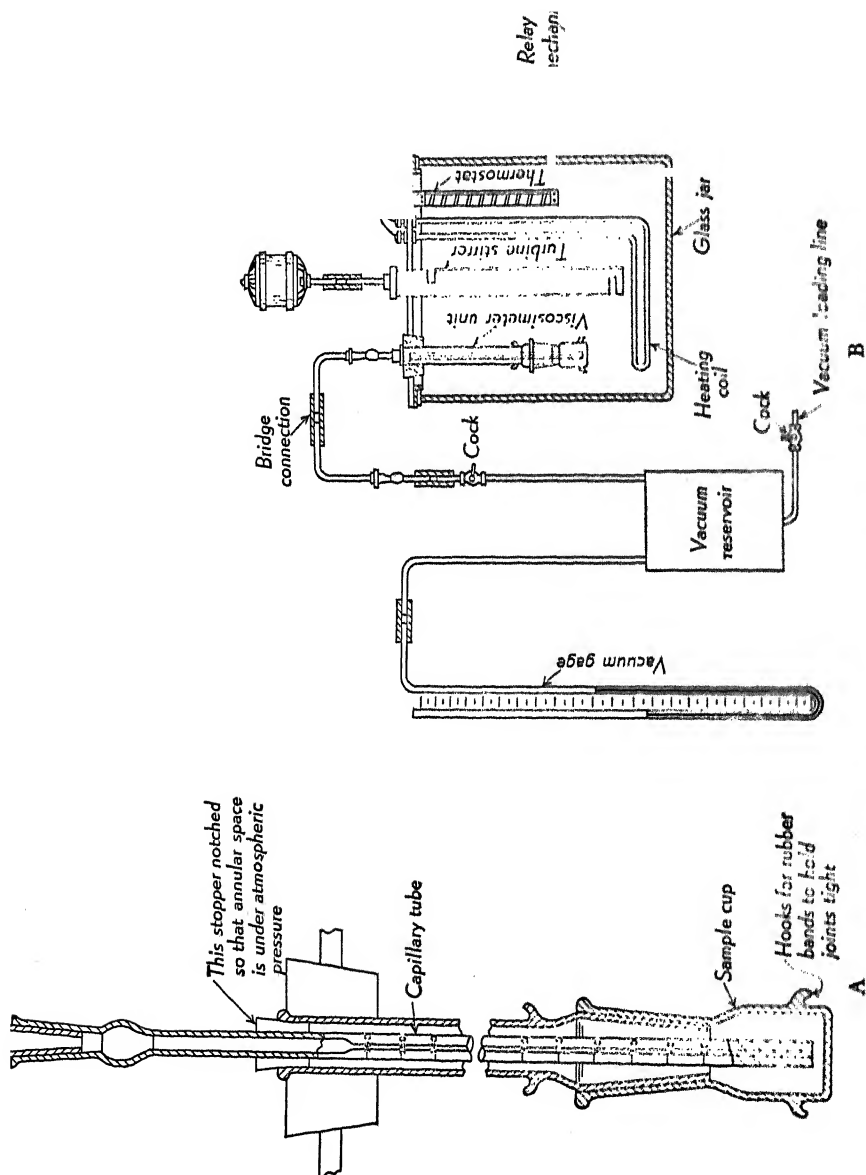


FIG. 233.—Apparatus for Determining Absolute Viscosity to Poises.

A—Cross section of Capillary-tube and Sample-cup; B—Assembly of Apparatus.

For fluid substances, start at the 40 mm. and end at the 120 mm. mark, using the following formula:

$$\mu = (25.909h - 17.163) r^2 t.$$

For more viscous substances, start at the 20 mm. and end at the 40 mm. mark, using the following formula:

$$\mu = (20.4h) r^2 t.$$

Where μ = absolute viscosity in poises; h = vacuum in cms. of mercury; r = radius of the capillary bore in cms.; and t = time of rise in seconds.

At the close of the test, the capillary tube may be cleansed with hot nitrobenzene, followed by acetone.

Method II: The following method has been standardized for determining kinematic viscosity:⁵⁶

This method is intended for determining the kinematic viscosity of any petroleum product or lubricant which is a true viscous liquid at the temperature of the test.

The apparatus shall be of the capillary type and shall be capable, under proper manipulation, of measuring viscosity with an error not greater than 0.2 per cent within the viscosity range in which it is to be used. Various types of apparatus have been used. Three types are shown in Fig. 234.

Two procedures for determining kinematic viscosity by means of apparatus conforming to the requirements prescribed. The procedure used shall give an accuracy equal to or greater than (not more than 0.2 per cent error) the methods referred to below.

Pure distilled water shall be the primary viscosity standard. The kinematic viscosity of water shall be taken as:

At 68° F. (20° C.)	1.007 centistokes
At 100° F. (37.8° C.)	0.689 centistokes
At 150° F. (54.4° C.)	0.518 centistokes

Oil samples may be used as secondary standards.

The proper suspended-level viscosimeter shall be selected. The viscosimeter shall be cleaned by rinsing twice with petroleum ether, benzol, or a narrow cut of naphtha. This solvent shall then be removed by passing a current of clean, dry air through the viscosimeter and for this reason the solvent used shall be of such volatility that it will be easily and completely removed under these conditions.

The viscosimeter shall be immersed in the bath so that bulb is below the surface of the bath liquid and shall be adjusted to a

vertical position. This may be accomplished by visual examination in two different vertical planes or by employing a small plumb bob consisting of a 1-cm. length of solder wire and a piece of silk thread attached to a perforated cork placed in the wide arm of the instrument. When properly aligned, the plumb bob will not touch the walls.

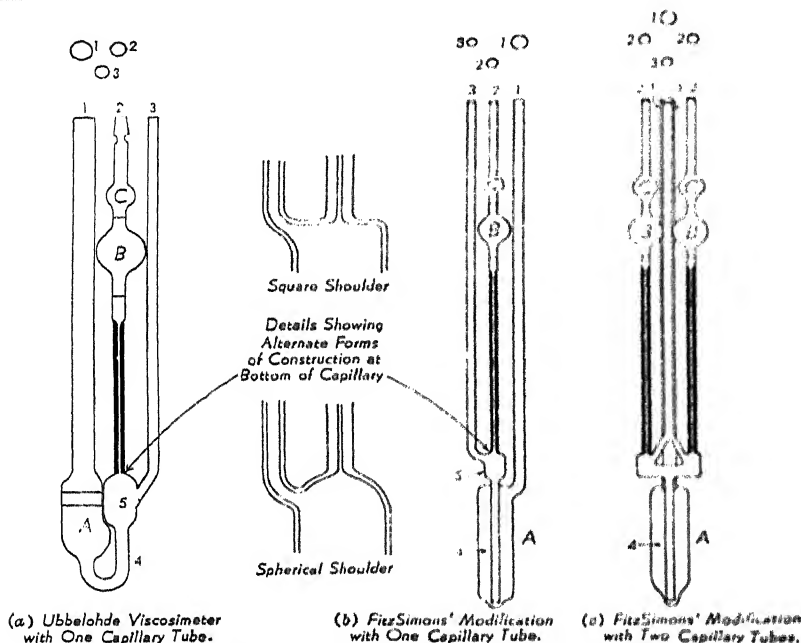


FIG. 234.—Suspended-level Viscosimeters for Kinematic Viscosity.

A small sample of oil (about 10 ml.) shall be filtered through a fritted (sintered) glass filter of medium porosity or a 100-mesh screen filter into a small beaker or bottle. The oil sample as well as the viscosimeter must be free from any solid particles, lint, etc. The oil sample shall be charged into tube 1 of the viscosimeter.

The bath shall be maintained at the temperature of test within $\pm 0.025^\circ \text{F}$. The viscosimeter shall remain in the constant-temperature bath long enough to reach the bath temperature. The minimum time required is 5 min. for the 100°F . (37.78°C .) bath, and 10 min. for the 210°F . (98.89°C .) bath.

After the sample has attained bath temperature, tube 3 shall

be stoppered with the finger and the oil shall be drawn into tube 2 by suction to some point above the upper mark but not above the center of the small bulb. (When using the double instrument, the tube 2 not being used must be closed as well as tube 3.)

The suction shall then be released and the finger removed from tube 3, allowing the oil to flow freely out of the capillary tube. The time in seconds required for the meniscus to pass from the upper to the lower mark shall be measured. If this efflux time is less than 80 sec., the next smaller viscosimeter shall be selected and the operation repeated. Determinations shall be repeated until two successive efflux times agree within 0.2 per cent, the average of these two determinations being used for calculating the kinematic viscosity.

The kinematic viscosity in centistokes shall be calculated from the following equation:

$$V = Ct \frac{B}{t}$$

where V the kinematic viscosity in centistokes,
 C the determined calibration constant for the instrument,
 t the efflux time in seconds, and
 B an experimental constant determined by the design of viscosimeter. For viscosimeters with capillary diameters of 0.75 mm. or larger, the value of this constant is 2.8 for the Ubbelohde design and 1.0 for the Fitz-Simons' design; in the case of viscosimeters having capillary diameters less than 0.75 mm., the value of the constant shall be determined.

Standard tables have been formulated for the conversion of kinematic into Saybolt universal and furol viscosities.⁵⁷

Method III: A modified form of viscosimeter for ascertaining the absolute viscosity consists of three capillaries of decreasing bore, fused together in alignment, with intervening bulbs. The coarsest (i.e., lowest) capillary measures a viscosity range from 1500 to 15,000 centistokes, the middle one from 150 to 1500 centistokes, and the upper one from 15 to 150 centistokes. A single temperature of 140° F. (60° C.) is recommended, and the instrument is claimed to have a range great enough to include all liquid road materials (i.e., from SC-0 to SC-6).⁵⁸ The use of the Höppler absolute viscosimeter has also been suggested.⁵⁹

Test 8cc. Hutchinson's Method. This is illustrated in Fig. 235. It was invented by John Hutchinson⁶⁰ and consists of a metal

spindle 9 in. long over all, bearing a conical-shaped disc (*C*) 2 in. in diameter, midway between the ends, with a plumb-shaped weight fastened to its lower end. The instrument is supplied with three weights (*D*) to be used with tars of different consistency or gravity. The spindle bears two rings (*A*) and (*B*), 2 in. apart.

The test is conducted by placing the bituminous material in a cylinder at least 9 in. high and 4 in. in diameter, filled to $\frac{1}{2}$ in. of the top. The bituminous material is brought to exactly 77° F., the tester introduced, and the time in seconds is noted for the spindle to sink from *a* to *b*. Weight No. 1 is recommended for tars having a specific gravity of 1.170–1.195, No. 2 from 1.195–1.215, and No. 3 from 1.215–1.240. The weights do not conform to standard weights or dimensions, and the instrument should not therefore be regarded as a strictly scientific one. It is used extensively in England.

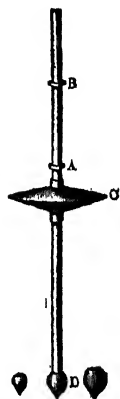


FIG. 235.—Hutchinson's Tar Tester.

Formulas and monograms have been worked out, showing the relation between the consistency and temperature of tars using the Hutchinson tester,⁶¹ also the relation between the consistency of tars and petroleum asphalts in various admixtures.⁶²

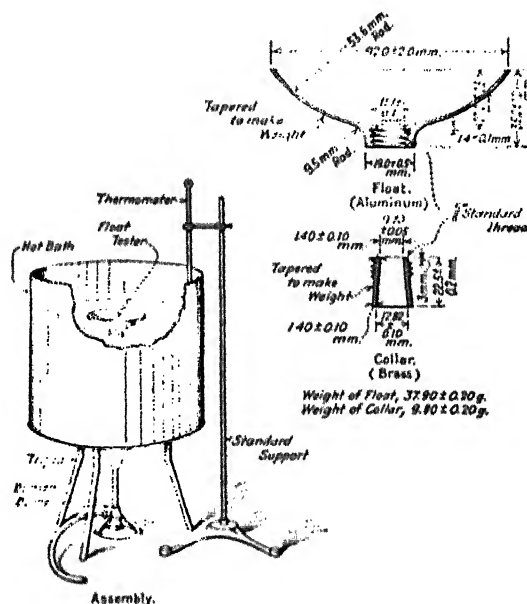
Test 8d. Float Test. This instrument is used largely for testing the viscosity or consistency of semi-solid bituminous materials. The range of the float test is limited, and it cannot be used with very fluid bituminous materials or with hard solids. It accordingly fills the gap between the Engler viscosimeter on one hand, and the needle penetrometer and consistometer on the other. The test is not affected by the presence of finely divided mineral matter or free carbon.

The procedure has been standardized as follows:⁶³

(a) The float (Fig. 236) shall be made of aluminum or aluminum alloy and shall be in accordance with the following requirements:

Weight of float, g.	37.70
Total height of float, mm.	34.0
Height of rim above lower side of shoulder, mm.	26.5
Thickness of shoulder, mm.	1.3
Diameter of opening, mm.	11.0

Minimum	Normal	Maximum
37.70	37.90	38.10
34.0	35.0	36.0
26.5	27.0	27.5
1.3	1.4	1.5
11.0	11.1	11.2



Courtesy A.S.T.M.

FIG. 216.—Float Test Apparatus.

(b) The collar shall be made of brass and shall be in accordance with the following requirements:

Weight of collar, g.	9.60
Over-all height of collar, mm.	22.3
Inside diameter at bottom, mm.	12.72
Inside diameter at top, mm.	9.65

Minimum	Normal	Maximum
9.60	9.80	10.00
22.3	22.5	22.7
12.72	12.82	12.92
9.65	9.70	9.75

The top of the collar shall screw up tightly against the lower side of the shoulder.

The assembled float and collar, with the collar filled flush with the bottom and weighted to a total weight of 53.2 g., shall float upon water with the rim 8.5 ± 1.5 mm. above the surface of the water. Dimensions of the apparatus additional to those required above are given in Fig. 236. The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being -2 to $+80^{\circ}$ C. or $+30$ to $+180^{\circ}$ F., respectively. The diameter of the bath and the depth of water shall be at least 185 mm.

The brass collar shall be placed with the smaller end on a brass plate which has been previously amalgamated with mercury by first rubbing it with a dilute solution of mercuric chloride or nitrate, and then with mercury.

The sample shall be completely melted at the lowest possible temperature that will bring it to a sufficiently fluid condition for pouring, excepting creosote oil residues, which shall be mixed and poured at a temperature of 100 to 125° C. It shall be stirred thoroughly until it is homogeneous and free from air bubbles. The sample shall then be poured into the collar in any convenient manner until slightly more than level with the top.

Asphalt and Asphalt Products. Asphalt and asphalt products shall be cooled to room temperature, placed in water maintained at 5° C. for five minutes, after which the surplus material shall be removed by means of a spatula, or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., $\pm 1^{\circ}$ C., and left in this bath for at least fifteen minutes.

Tar Products. Tar products shall be immediately immersed in ice water maintained at 5° C. for five minutes, after which the surplus material shall be removed by means of a spatula or steel knife, which has been slightly heated. The collar and plate shall then be placed in a tin cup containing ice water maintained at 5° C., $\pm 1^{\circ}$ C., and left in this bath for at least fifteen minutes.

The bath shall be filled with water and the water heated to the temperature at which the test is to be made. This temperature

shall be accurately maintained and shall at no time throughout the test be allowed to vary more than 0.5° C. from the temperature specified.

After the material to be tested has been kept in the ice water for not less than fifteen minutes nor more than thirty minutes, the collar with its contents shall be removed from the plate and screwed into the aluminum float and immersed in water at 5° C. for one

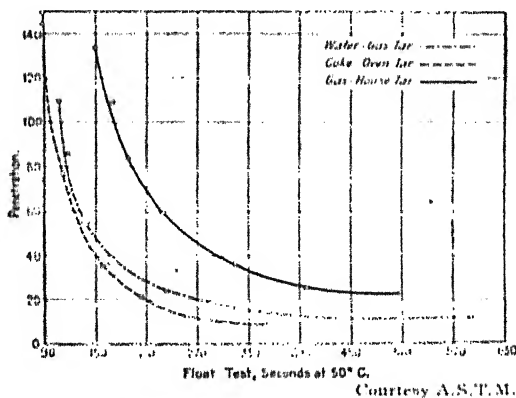


FIG. 237.—Comparison of the Float and Penetration Tests for Different Types of Tars.

minute. Any water shall then be removed from the inside of the float and the latter immediately floated in the warm bath. As the plug of material becomes warm and fluid, it is forced upward and out of the collar until the water gains entrance into the saucer and causes it to sink.

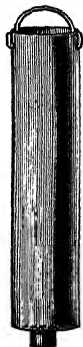
The time in seconds between placing the apparatus on the water and when the water breaks through the material shall be determined by means of a stop watch, and shall be taken as a measure of the consistency of the material under examination.

NOTE.—Special precautions should be taken to insure the collar fitting tightly into the float and to see that there is no seepage of water between the collar and float during the test.

Figure 237 shows the relation between the float test and penetration test (200 g., 5 sec., 32° F.) of three types of tars, all of which were evaporated and samples taken having softening points

of 95, 100, 105, 110, 115, 120 and 125° F. respectively, cube-in-water method:⁶⁴

The author has also found the float test of value for testing bituminous substances at a temperature *exactly* 50° F. above the fusing-point by the R. and B. method, thereby furnishing a criterion of the susceptibility to temperature changes, also a means of distinguishing between blown and residual asphalts.



Courtesy of A. H. Thomas Co.

FIG. 238.—Schutte Viscosity Tester.

Test 8e. Schutte Method. This instrument, as illustrated in Fig. 238, operates similarly to the float tester, with the difference, however, that the pressure is applied by a column of water above the plug of pitch. The melted bituminous material is first introduced into a brass collar 1 in. high and $\frac{5}{8}$ in. in diameter. This is placed in water at the required temperature for at least ten minutes, and then screwed into the tube ($10\frac{1}{8}$ in. long). The apparatus is immersed in water maintained at the required temperature so the water level just covers the lower shoulder of the tube, which is then completely filled with

water at the given temperature, and the time interval recorded between the filling of the tube and the displacement of the plug of bituminous material at the bottom. Check tests are said to agree within five seconds.⁶⁵

Test 8f. Falling Ball Method. An electrical method has been devised⁶⁶ for recording the time in which a spherical steel ball falls a definite distance through the material at a given temperature, which is accomplished by noting the alterations in inductance and capacity of a closed oscillatory circuit of 31.5 megacycles, by the passage of the ball through two "search coils" in the circuit, suspended at suitable positions in the material under examination. This method has been shown to yield scientifically correct results in absolute units, and will indicate whether or not the substance is a true liquid at a given temperature.⁶⁷ The apparatus is illustrated diagrammatically in Fig. 239, in which "b" represent an oil-bath; "V" the viscosimeter tube composed of an aluminum alloy, of an in-

ternal diameter sufficient to avoid "wall effect" when using the largest ball ($\frac{1}{2}$ in. diam.); "C" the search coils spaced exactly 10 cm. apart (center to center), the upper coil being approximately 10 cm. from the surface of the liquid and the lower coil approximately 10 cm. from the bottom of the viscosimeter tube; "H" the entrance hole for introducing the spherical ball; "T" the terminals; and "S" the stirrer. The search coils are wound in units of about 1 in. diameter and $\frac{7}{16}$ in. long, and are mounted on a skeleton copper tube frame through which the lead wires are passed. They are wound in thin paper, thoroughly coated with shellac and baked. Audible indication of the passage of the falling ball through the search coils is obtained by coupling the circuit to a similar circuit tuned to produce audible notes on a telephone receiver. Balls ranging in diameter from $\frac{1}{16}$ in. to $\frac{1}{2}$ in. may be used and tests may be carried out at any desired temperature.

The viscosity in poises may be calculated from the following formula:

$$\eta = \frac{td^3(S-s)g}{18c(1-2.4d/D)(1-5d/3h)}$$

where η = absolute viscosity in poises,

D = diameter of the viscosimeter tube,

d = diameter of the sphere,

S = density of the sphere in grams per ml.,

s = density of the substance in grams per ml.,

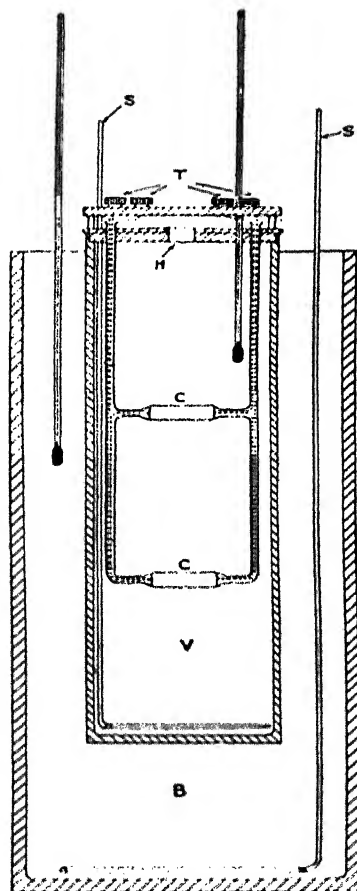
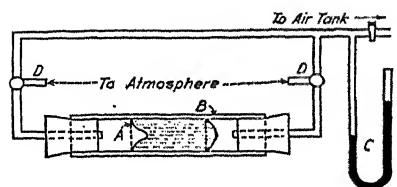


FIG. 239.—Falling-ball Viscosimeter.

c = distance in cm. between the two search coils,
 h = height of the test column of substance in cm.,
 t = time in seconds for the sphere to drop the distance " c "
 between the two coils,
 $g = 981 \text{ cm./sec.}^2$

The viscosity of hard pitches and asphalts may also be measured by pressing a steel sphere against the flat surface for a given time with a given force, and measuring the diameter of the indentation.⁶⁸

Test 8g. Alternating Stress Method. This procedure is adapted to testing the viscosity of semi-solid bituminous substances



Courtesy A.S.T.M.

FIG. 240.—Apparatus Used in Alternating Stress Method.

in absolute units⁶⁹ ranging from about 1,000,000 to 100,000,000 poises. The apparatus is illustrated diagrammatically in Fig. 240, and consists of a cylinder "A" of the material, about 5 cm. long, contained in a glass tube "B" which may range in diameter from about 0.25 to 2.00 cm., connected to a tank of compressed air and a manometer "C" by three-way stopcocks "D," so

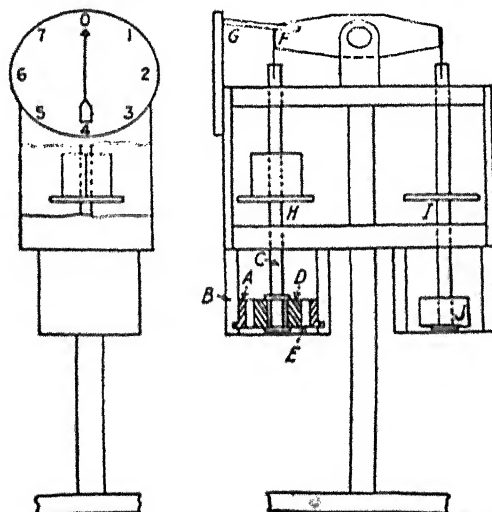
that pressure may be applied to either end of the cylinder of bituminous material. The material is introduced in the tube "B" while held in a vertical position with an amalgamated brass plunger having a flat end inserted part way into the tube. The melted substance is then poured into the tube and onto the plunger, and upon cooling, both ends of the plug are flattened by manipulation with the amalgamated plunger. The tube and its contents are placed in a constant temperature bath so that the ends of the cylinder "A" may be observed by means of a cathetometer. The cathetometer is focused on one end of the cylinder and measured air pressure applied to the other end. An extrusion of the material occurs, the rate of movement being measured by the cathetometer. When the extrusion takes place to a distance equal to one-half of the radius of the tube, it is forced back by reversing the direction of the air pressure. At first, high pressures are applied and each

succeeding pair of determinations is made at progressively lower pressure until a constant reading is obtained. If the substance is elastic or thixotropic, the sample must be worked back and forth several times before a reading is taken. Viscosity in poises may be calculated from the formula:

$$\eta = \frac{PR^2t}{4Lh}$$

where η viscosity in poises,
 P pressure applied in dynes per sq. cm.,
 R radius of the tube in cm.,
 t time of flow in seconds,
 L length of the cylinder in cm., and
 h length of extrusion in cm., in the time " t ."

Test 8h. Falling Coaxial Cylinder Method. This method is likewise adapted to measuring the viscosity of semi-solid bituminous substances ranging from 5,000 to 100,000,000 poises.⁷⁰ A schematic diagram of the apparatus is illustrated in Fig. 241, and consists of a brass outer cylinder "A" measuring $\frac{3}{4}$ in. in diameter;



Courtesy A.S.T.M.

FIG. 241.—Falling Coaxial Cylinder Method.

held in a bakelite holder "B" by arrangement which permits rapid insertion or removal of the mold. A bakelite rod "C" passes through the inner cylinder "D" measuring $\frac{1}{2}$ in. in diameter and 1 in. in length. The material under test fills the annulus "E" measuring 16 ml. in volume. The bakelite rod "C" is connected to a metal ribbon which passes over a rocker-arm "F," which in turn is attached to another rod "I" and a counterpoise "J." A weight "H" applied to the rod "C" causes the inner cylinder "D" of the viscosimeter to move downward; while added to rod "I" it causes cylinder "D" to move upward, the distance of movement of the inner cylinder in a given time being indicated on the dial "G." The apparatus is equipped with a series of weights to measure a wide range of viscosities. A thermostatically controlled bath surrounds the coaxial cylinders and the holder "B." Rod "C" and holder "B" are made of a non-metallic material to eliminate the conduction of heat away from the sample. The two cylinders "B" and "D" are heated and placed on an amalgamated brass plate which is provided with pins to center both cylinders, whereupon the melted substance is poured into the annular space, and when cool the base plate is removed and the apparatus assembled. After the inner cylinder has moved a predetermined distance, as measured by a cathetometer, the apparatus is reversed and the inner cylinder allowed to return to its original position. A test usually consists of three "out" and three "in" readings. The viscosity is calculated from the following formula:

$$\eta = \frac{gt}{2\pi Lh} \left[\log_e \frac{R}{r} (W - \pi r^2 LD) + \frac{\pi LD}{2} (R^2 - r^2) \right]$$

where η = viscosity in poises,

g = gravitation constant,

L = length of outer cylinder in centimeters,

h = distance in centimeters of movement in time t (sec.),

R = inner radius of outer cylinder in centimeters,

r = radius of inner cylinder in centimeters,

W = effective weight in grams of applied load (actual weight minus buoyant effect of liquid bath), and

D = difference in density of material being measured and density of bath in which the apparatus is run in grams per cubic centimeters.

If the bath in which the viscosimeter is immersed contains a liquid of substantially the same density as the substance being tested, the equation simplifies to:

$$\eta = \left(\log_r \frac{K}{r} \right) \left(\frac{g}{2\pi L} \right) \left(\frac{Wt}{h} \right) = KH' \frac{t}{h}$$

where K is constant for a particular instrument. For thixotropic materials, the first "out" reading will give an abnormally low reading, but all subsequent readings will approach a constant value. On the other hand, if the material is highly elastic, the initial rate of movement when the direction of flow is reversed, will be high. These low and high readings should be discarded when calculating the viscosity of the material.

The falling coaxial cylinder viscosimeter has also been utilized for ascertaining the consistency of asphalts carrying various types of mineral fillers.⁷¹

Test 8i. Rotating Cylinder Viscosimeter.⁷² This type of tester is similar to the falling coaxial cylinder described in Test 8h, and may be used for determining high consistencies, ranging from 0.001 to 1000 megapoises. The apparatus consists of a rotating cylinder of the form illustrated diagrammatically in Fig. 242. The test is carried out in the following manner:

The viscosimeter proper is shown in Fig. 242. The space, D , between the rotor, A (outer chamber), and stator, B (inner cylinder), is filled with the material to be tested. As the outer chamber rotates at a constant angular velocity, the torque required to prevent B from rotating is measured. The top and bottom of the stator are cones of such an angle that the mean rate of shear is essentially the same at the ends as in the cylindrical part of the annulus. The lid, C , has the advantage of preventing elastic asphalts from pulling away from the inner cylinder under the shearing action.

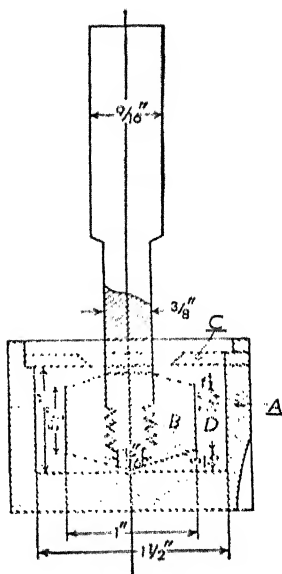


FIG. 242.—Diagram of Rotating Cylinder Viscosimeter.

The brass surface of *B* and the inner surface of *A* are knurled in order to minimize slippage at the metal-liquid interface when materials of high consistency are being tested.

The cylinder is driven by a constant-speed motor, connected through a gear reducer, and a series of gears capable of being shifted readily in order to obtain different angular velocities for the rotor. The torque required to keep the stator from rotating varies, depending on the consistency of the material being tested and the rate of shear employed. A range of torque is covered by using weights of various sizes at different distances from the center. The viscosimeter is calibrated to measure the deflection of the indicator which is directly proportional to the consistency.

The viscosity or consistency is calculated by the equation:

$$\eta = K(T/\omega)$$

where η = viscosity or consistency, poises,

T = torque required to prevent the stator from turning,
dyne-centimeters,

ω = angular velocity of rotor, radians per second,

K = a constant determined by the dimensions of the viscometer, cm.^{-3}

HARDNESS OR PLASTICITY

This constitutes one of the most important tests for examining bituminous materials, and is employed for purposes of identification, considered either alone or in conjunction with the fusing-point; for determining the adaptability of bituminous materials in connection with certain proposed uses; for gauging the uniformity of supply; and for purposes of factory control.

The terms "consistency," "plasticity," etc., have been defined as follows:⁷³

Consistency.—That property of a body by virtue of which it tends to resist deformation.

Plasticity.—That property of a body by virtue of which it tends to retain its deformation after reduction of the deforming stress to its yield stress.

Elasticity.—That property of a body by virtue of which it tends to recover its original size and shape after deformation.

Liquid.—A substance which undergoes continuous deformation when subjected to shearing stress.

Simple Liquid.—A liquid in which the rate of shear is proportional

to the shearing stress. The constant ratio of shearing stress to rate of shear of a simple liquid is the *viscosity* of the liquid.

Complex Liquid.—A liquid in which the rate of shear is not proportional to the shearing stress.

Solid.—A substance which undergoes permanent deformation only when subjected to shearing stress in excess of some finite value characteristic of the substance (yield stress).

Plastic Solid.—A substance which does not deform under a shearing stress until the stress attains the yield stress, when the solid deforms permanently.

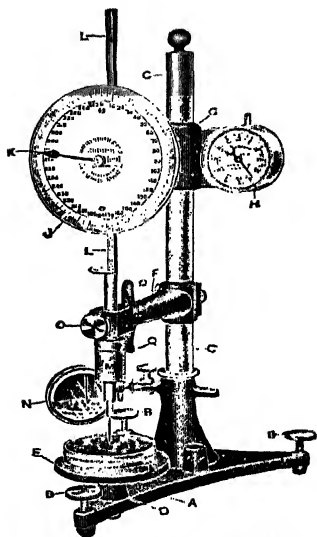
Elastic Solid.—A substance in which, for all values of the shearing stress below the rupture stress (shear strength), the strain is fully determined by the stress, regardless of whether the stress is increasing or decreasing.

Test 9a. Moh's Scale. This test has long been used for recording the hardness of minerals by comparing their resistance to abrasion with substances of known hardness. Ten minerals are used in a graduated scale of units, viz.: (1) talc, (2) gypsum, (3) calcite, (4) fluorite, (5) apatite, (6) orthoclase, (7) quartz, (8) topaz, (9) sapphire and (10) diamond. A pointed fragment of the standard mineral is moved back and forth several times on the same line, a short distance across the surface of the bituminous material under test. If the bituminous material is not scratched, it is harder than the mineral used, whereas if it is scratched, it may be either softer or of the same hardness as the standard mineral. If it is of the same hardness, it will in turn scratch the surface of the standard mineral but if it is softer, it will have no effect. The first four standard minerals are used for this purpose, as the hardest bituminous materials encountered usually do not test higher than 4 on Moh's scale.

Test 9b. Penetrometer. This was originally devised by H. C. Bowen in 1888.⁷⁴ The first crude instrument was further improved by A. W. Dow.⁷⁵ The Dow penetrometer as simplified in construction by Clifford Richardson and C. N. Forrest represents the type in use today,⁷⁶ both forms operating on the same principle and giving the same readings.

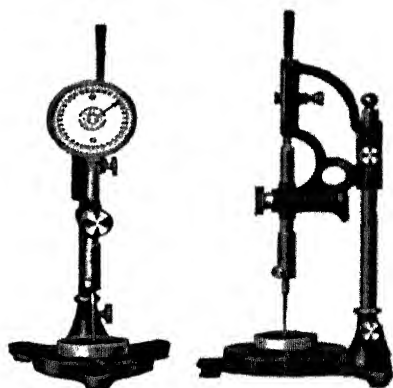
The Richardson-Forrest improved penetrometer is illustrated in Fig. 243. The base *A* may be levelled by the thumb-screws *B*,

and is attached to the standard *C* and also the platen *D*, which by means of a screw-shank raises or lowers the revolving disc *E*, on which is placed the sample of bituminous material to be tested. The standard *C* carries a bracket *F* adjustable as to elevation by a thumb-screw, also the bracket *G*, which on the back carries the clockwork *H* timing the duration of the test by half-second beats,



Courtesy of Precision Scientific Co.

FIG. 243.—Standard Penetrometer.



Courtesy of Precision Scientific Co.

FIG. 244.—Miniature Penetrometer.

and on the front the dial *J* divided into 360 degrees, with the hand *K* marking the number of degrees, each of which represents one-tenth millimeter of penetration measured by rack on sliding gauge *L*, engaging in pinion on the shaft which actuates the hand *K*. The bevelled-edge mirror *N*, adjustable through universal joints, serves to reflect light on the sample under test. The plunger *O* acts as a brake, which holds the needle bar, representing a weight of 50 g. together with the superincumbent weight in place, until pressed inward, which movement permits the needle and weight to act upon test-block without friction, and is easily operated by grasping the horns *Q* between two fingers and pressing the brake-head *O* with

the thumb. *M* represents a weight of predetermined capacity, either 50 or 150 g.

Forms of penetrometer operated by automatic timing devices have also been constructed.¹⁷ A miniature penetrometer for portable use is illustrated in Fig. 244, and a form with a micrometer measuring device is illustrated in Fig. 245.

An ingenious automatic penetrometer has been devised by John Hutchinson⁷⁸ as illustrated in Fig. 246 (A), (B), (C), and (D), which operates in the following manner:

The timing mechanism is enclosed in a dust-proof "head" and the principle of operation is illustrated in the illustration of the "timing head," Fig. 246 (B), and the diagram, Fig. 246 (C). Normally, the beam is locked in position by the spring-loaded lever *L* which carries a friction pad bearing on the back of the beam. If required, during preliminary adjustment, the beam may be brought to a setting position by sliding it against this frictional lock. The other end of the lever *L* carries a short pin *P* which rides over the surface of a brass cam *C*. This latter is circular but has a slot *S* cut in the circumference into which the pin *P* may drop once per revolution.

When the pin is riding on the surface of the cam the brake lever *L* is held away from the penetrometer beam *B* and the latter is free to move vertically through the edge bearings *E* and *E*1. Upon entry of the pin into slot *S* the lever *L* is allowed to move forward and lock the beam.

The cam is driven, through a chain of pinions, from a driving sprocket *K* which is impulsed by a short, stout driving spring *D*—the driving sprocket being loaded by depressing the external lever *A*. The cam wheel also drives, through a second train of pinions, a high speed escapement *T* which accurately controls the speed of rotation of the cam wheel. Upon releasing the beam by pressing

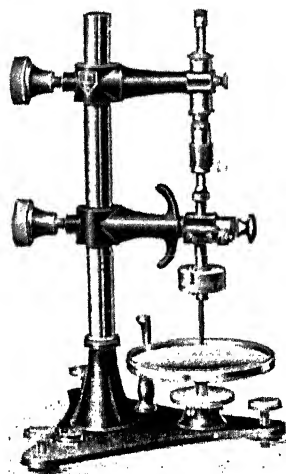


FIG. 245.—Penetrometer with
*Micrometer Attachment.

the external trigger *R* the energy stored in the driving sprocket rotates the cam at a constant speed which is such that one revolution brings the slot into the engagement with pin *P* in exactly five seconds, thus again locking the beam.

The vertical movement of the beam may be read by means of the vernier gear *V*. A zero setting of the latter is obtained by

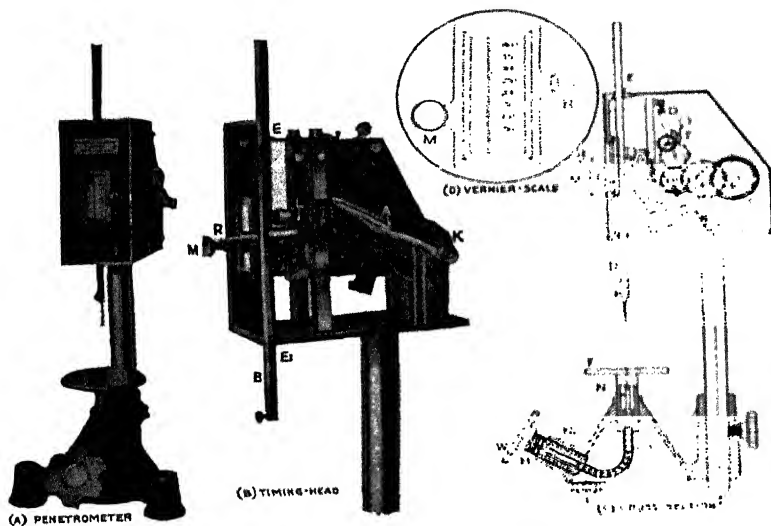


FIG. 246.—Hutchinson Automatic Penetrometer.

means of the adjusting knob *M*. Penetration is therefore easily read directly in 1/100 cm. without the need of a rack and pinion operated dial.

The sample platform, illustrated in Fig. 246 (C), incorporates a new method of elevation which allows very rapid but accurate adjustment of the sample surface to "scratch contact" with the needle point. The chromium plated table *F* is carried in a vertical bearing machined in the base casting, and is fitted with a plunger *N* sliding in a metal tube. This latter carries a chain of ball bearings *G* which, at the other end of the tube, bear against a second plunger *N1*. This is concentrically attached to the operating hand wheel *W* which rotates in the screw thread *H*. Thus, upon ro-

tating the hand wheel the movement is transmitted to the table *F*, raising or lowering it rapidly but with absolute precision. This mechanism confers all the advantages of a hydraulic system but removes the possibility of leakage of oil or operating fluid. The inconvenience attached to the rotation of a screw-mounted table is also obviated but the platform is capable of rotation, if necessary, during final adjustment.

Assuming that the sample has been adjusted accurately to "scratch contact" with the needle, the vernier is set to zero relative to the graduations on the beam. The lever, situated on the right hand side of the head, is then depressed to the full extent. Upon pressing the operating trigger, situated to the right of the vernier window, the beam will be released but automatically locked again after a five second period. The penetration may then be read directly from the relative position of the beam graduations and those on the vernier.

The principles underlying the vernier are very well known, but a diagram of the gear in this instrument is shown in Fig. 246 (D). The vernier indicates a penetration of 64° .

(1) The instrument gives absolutely accurate timing of the penetration period thus eliminating possible errors due to manual operation with the aid of a stop watch. The high speed escapement controls the penetration period to an accuracy of $\frac{1}{10}$ sec.

(2) It is robust and contains a minimum of moving parts subject to displacement or wear. All operating mechanism is enclosed in a dustproof case.

(3) Friction on the penetrometer beam is extremely small because the bearing surfaces are very short and well spaced. It is impossible for the beam to become jammed or to rock sideways—a defect which is common on some machines using a single, long bearing.

(4) The sample platform lifting gear enables rapid and precise adjustment to be made. The mechanism is enclosed in a heavy base casting and cannot jam or become unstable.

(5) The vernier system of reading penetrations eliminates the rack-and-pinion operated pointer and dial which is very subject to wear. The method gives direct reading in penetration degrees ($\frac{1}{100}$ cm.).

Careful investigations have been made as to the diameter of the holder for the bituminous material;⁷⁹ the method of preparing the

specimen;⁸⁰ the size and shape of the needle;⁸¹ also other variable factors.⁸² As a result of these, the following standard tests have been adopted.

*I. For Asphalts and Pitches.*⁸³ Penetration is defined as the consistency of a bituminous material, expressed as the distance that a standard needle vertically penetrates a sample of the material under known conditions of loading, time and temperature. Where the conditions of test are not specifically mentioned, the load, time and temperature are understood to be 100 g., 5 sec., 25° C.

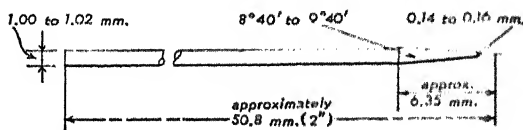


FIG. 247.—Needle for Penetration Test.

(77° F.), respectively, and the units of penetration to indicate hundredths of a centimeter.

A container, in which the sample is tested, made of metal or glass, cylindrical in shape, and having a flat bottom shall have the following inside dimensions: 55 mm. (2.17 in.) in diameter, and 35 mm. (1.38 in.) in depth.

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

The needle (Fig. 247) for this test shall be made from a cylindrical steel rod approximately 50.8 mm. (2 in.) long, and having a diameter of 1.00 to 1.02 mm. This shall be symmetrically tapered at one end to a cone approximately 6.35 mm. ($\frac{1}{4}$ in.) in height and whose angle shall be within the range of 8° 40' and 9° 40'. After tapering, the point shall be "blunted" by grinding off to a truncated cone, the smaller base of which shall be from 0.14 to 0.16 mm. in diameter. The finished needle shall be hardened and highly polished.

The water bath shall be maintained at a temperature not varying more than 0.1° C. from 25° C. (77° F.). The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm. (4 in.) and shall be supported on a perforated shelf not less than 5 cm. (2 in.) from the bottom

of the bath. Any apparatus which will allow the needle to penetrate without appreciable friction, and which is accurately calibrated to yield results in accordance with the definition of penetration, will be acceptable.

The transfer dish for container shall be a small dish or tray of such capacity as will insure complete immersion of the container during the test. It shall be provided with some means which will insure a firm bearing and prevent rocking of the container.

The sample shall be completely melted at the lowest possible temperature and stirred thoroughly until it is homogeneous and free from air bubbles. It shall then be poured into the sample container to a depth of not less than 15 mm. ($\frac{5}{8}$ in.). The sample shall be protected from dust and allowed to cool in an atmosphere not lower than 18° C. (65° F.) for one hour. It shall then be placed in the water bath along with the transfer dish and allowed to remain one hour.

In making the test, the sample shall be placed in the transfer dish filled with water from the water bath of sufficient depth to completely cover the container. The transfer dish containing the sample shall then be placed upon the stand of the penetration machine. The needle loaded with specified weight shall be adjusted to make contact with the surface of the sample. This may be accomplished by making contact of the actual needle-point with its image reflected by the surface of the sample from a properly placed source of light. Either the reading of the dial shall then be noted or the needle brought to zero. The needle is then released for the specified period of time, after which the penetration machine is adjusted to measure the distance penetrated.

At least three tests shall be made at points on the surface of the sample not less than 1 cm. ($\frac{3}{8}$ in.) from the side of the container and not less than 1 cm. ($\frac{3}{8}$ in.) apart. After each test the sample and transfer dish shall be returned to the water bath and the needle shall be carefully wiped toward its point with a clean, dry cloth to remove all adhering bitumen. The reported penetration shall be the average of at least three tests whose values shall not differ more than four points between maximum and minimum.

When desirable to vary the temperature, time and weight, and in order to provide for a uniform method of reporting results when

variations are made, the samples shall be melted and cooled in air as above directed. They shall then be immersed in water or brine, as the case may require, for one hour at the temperature desired. The following combinations are suggested:

At 0°C. (32°F.) 200-g. weight, 60 sec.

At 46.1°C. (115°F.) 50-g. weight, 5 sec.

When three penetrations at three different temperatures, all ascertained with a given weight acting for a fixed time period, are known, it is possible accurately to plot the penetration-temperature curve of the particular specimen of bituminous substance," by means of the following equation:

$$p = A + (BC)^t$$

where p = penetration in decimillimeters; t = the temperature in $^{\circ}\text{C.}$; and A , B and C = constant characteristics of the particular substance under examination. If the three temperatures in $^{\circ}\text{C.}$ are so chosen that:

$$t_3 - 2t_2 + t_1 = 0$$

(e.g., 50° , 25° , and 0°C. , or else 40° , 20° and 0°C.), where 0°C. is taken for t_1 , then the first stated equation will assume the following simple form:

$$\frac{p_3 - p_2}{p_2 - p_1} = C^{t_2}$$

By plotting " $\log (p - A)$ " against " t ", a *straight line* is obtained for any particular substance, from which it is possible to calculate or read off the constants B and C . The slope of the straight line represents " $\log C$ " and its intercept with the ordinate equals " $\log B$." It should be noted that B is a dimensional constant, which by being suitably modified will enable any temperature scale to be employed.

It has also been shown that if the time of penetration is maintained constant, there exists a fixed relation between weight and depth of penetration at varying temperatures. In all cases, the ratio of two penetrations obtained with two different weights remains practically constant over the entire temperature range. This

fact may be utilized whenever it is desirable to measure penetration with weights either smaller or greater than 100 grams.

A further modification consists in maintaining the time (5 sec.) and depth of penetration (0.04, 0.15, or 0.30 cm.) constant for a given temperature of test (32, 77, or 115° F.), and varying the weight required to force the standard needle into the specimen to a specified depth in the specified time. A range of weights from 0.1 to 600 g. is used for this purpose. At least three penetration measurements are made with different weights, so that at least one penetration is above and one below the specified depth, whereupon the results are plotted, and the weight required to give the specified penetration is obtained by interpolation. This procedure is particularly suitable for examining specimens in thin layers, as for example exposure panels in accelerated weathering tests, in which case the depth of penetration is maintained at 0.04 cm., as the thickness of the layer ranges from 0.0635 to 0.076 cm. Since the hardness determinations require only a few penetration measurements on each panel, the test specimen may be regarded as practically undisturbed in conducting further weathering tests.⁸⁵

II. For Greases. The following modification is used for testing the penetration of greases and of petrolatum:⁸⁶

This method of test is intended for use in measuring with a penetrometer the unworked or the worked consistency of lubricating greases which have a worked consistency less than 400, and in measuring the original consistency of petrolatum.

The apparatus shall consist of the following:

Penetrometer. A suitable penetrometer, with special cone, which permits the cone to drop without appreciable friction and which indicates accurately the depth of penetration. A simplified sketch of such a penetrometer with cone attached is shown in Fig. 248. The scale of the penetrometer shall be calibrated in tenths of a millimeter.

Cone. A cone, constructed of stainless steel or brass, with a detachable hardened-steel or stainless-steel tip. It shall conform to the dimensions shown in Fig. 249 except that the interior construction may be modified as desired. The outside surface of the cone and tip shall be given a very smooth finish. The total moving weight of the cone and attachments shall be 150 g.

Grease Worker. A worker, as shown in Fig. 250, to be used when the worked consistency of lubricating grease is to be measured.

Constant-temperature Bath. A constant-temperature bath regulated to $77^{\circ} \pm 1^{\circ}$ F. ($25^{\circ} \pm 0.5^{\circ}$ C) is desirable to bring the samples to the temperature of test, if many tests are to be made.

Lubricating Grease. Tests for the unworked consistency of a lubricating grease shall be made only on a sample in the original container (or on a cake or slab in the case of very hard

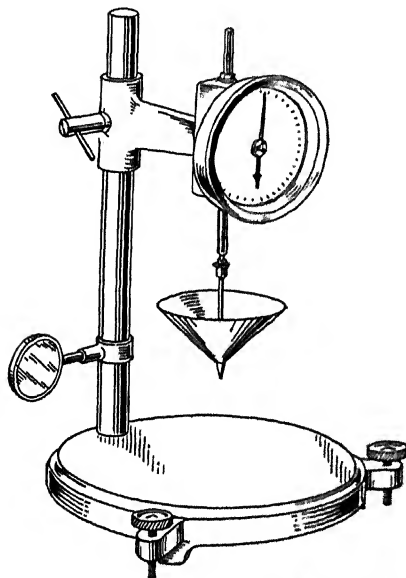
greases). Cans having a capacity of 1 lb., approximately 8 cm. in diameter, are the best containers for this purpose. The penetration of soft lubricating greases will vary with the diameter of the container. If samples are taken from large containers, more or less working is involved; consequently, the test shall be made only on a sample, worked as specified. Samples of discolored or rancid grease shall be rejected.

Petrolatum. All samples of petrolatum shall be tested for original consistency after melting and cooling to the temperature of the test.

Several samples will be needed when soft lubricating greases or petrolatum are tested.

(a) *Unworked Consistency of Lubricating Grease and Petrolatum.* The temperature of the sample shall be brought to

$77^{\circ} \pm 1^{\circ}$ F. ($25^{\circ} \pm 0.5^{\circ}$ C.) before the test. If the sample is initially within 3 to 4° F. (1.5° to 2° C.) of this temperature, it may be brought to 77° F. (25° C.) by placing in a water bath for 30 or 40 min.; but if the initial temperature is outside this range, it shall be placed in the constant-temperature bath for $1\frac{1}{2}$ hr. to obtain the desired constant temperature. If the room temperature is more than 3 to 4° F. (1.5° to 2° C.) from 77° F. (25° C.) a lid shall be placed on the can, sealing throughout with grease or petrolatum to prevent the entrance of water, and the can immersed in the bath for the required period. Otherwise, the temperature of the surface will be different from that of the main body of the lubricating grease or petrolatum.



Courtesy A.S.T.M.

FIG. 248.—Penetrometer for Testing Grease.

The surface of the sample of lubricating grease or petrolatum shall be cut level and very smooth with a knife. Care shall be taken not to work the surface.

The can of lubricating grease or petrolatum shall be placed on the penetrometer table and the cone lowered until the tip just touches the top surface of the sample. Watching the shadow of the tip

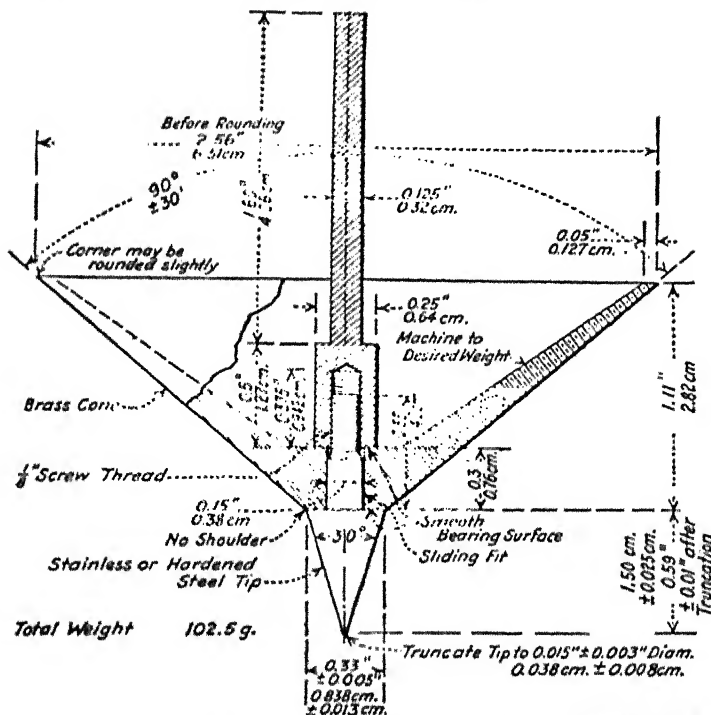
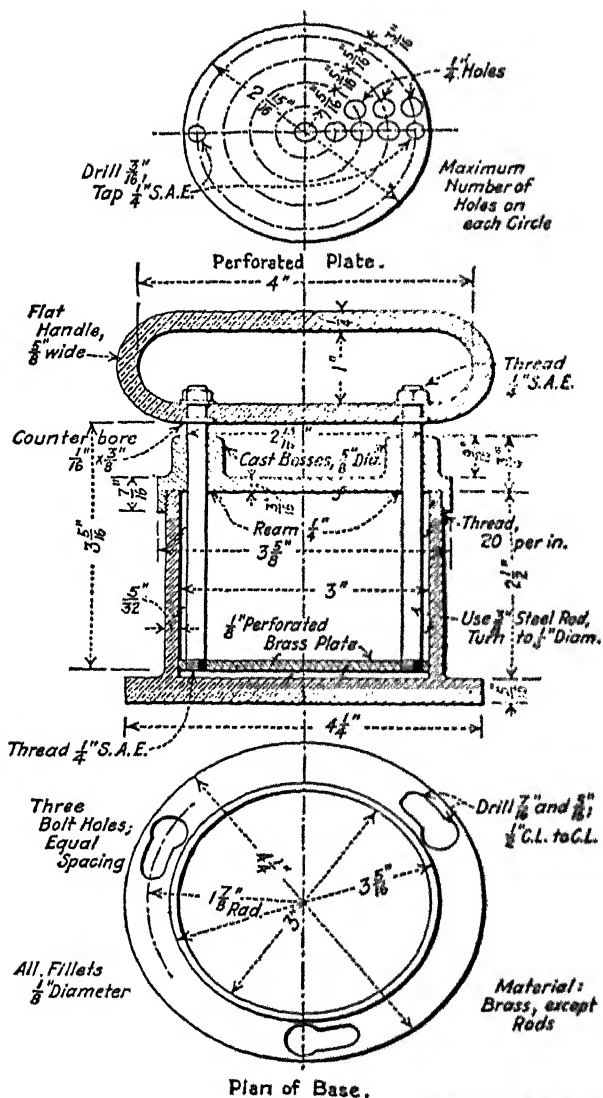


FIG. 249.—Penetrometer Cone.

Courtesy A.S.T.M.

is an aid to accurate setting. When testing soft lubricating greases (unworked consistency of 310 or more), it is very important that the cone tip shall be placed as exactly as possible at the center of the lubricating grease container. With older penetrometers it is then necessary to set the scale at zero, but with new apparatus the scale and cone move as a unit and no setting is needed. Finally, the plunger shall be quickly released and held free for 5 sec. The total penetration shall be read from the scale and reported as the unworked consistency.



Courtesy A.S.T.M.

FIG. 250.—Grease Worker.

The total surface area disturbed by the test will have a diameter about equal to the measured depth of penetration. In order to prevent one test from being affected by the disturbed area of a previous test or by sides of the can, the tip shall never be placed nearer the sides of the can or the edge of a previous hole than the penetration distance of that particular lubricating grease or petrolatum. The lubricating grease or petrolatum shall not be smoothed over for further tests.

Five tests shall be made and the average reported as the unworked consistency if the mean deviation of these readings does not exceed 3.0 per cent. If the mean deviation exceeds 3.0 per cent, the average of ten readings shall be reported.

(b) *Worked Consistency of Lubricating Grease.* The procedure for determining the consistency of worked lubricating greases (Note 1) shall be the same as that for unworked consistency except that the sample to be tested shall be transferred to the worker, which shall be filled heaping full with a minimum inclusion of air, brought to $75^{\circ} \pm 2^{\circ} \text{ F.}$ ($24^{\circ} \pm 1^{\circ} \text{ C.}$) and worked with 60 full double strokes of the plunger (Note 2). The top and plunger shall then be removed (Note 3) and the temperature adjusted to $77^{\circ} \pm 1^{\circ} \text{ F.}$ ($25^{\circ} \pm 0.5^{\circ} \text{ C.}$). Then the surface shall be smoothed and the test made as described. As soon as one test is finished, the surface may be smoothed over for the next, taking care to avoid creating air pockets.

NOTE 1.—Due to the rapid change in consistency of cold-set lubricating greases after working it is not advisable to determine their worked consistency.

NOTE 2.—In working stiff greases a wall bracket for holding the worker is helpful. Metal strips open on the inside are welded on three sides of a steel plate 6 by 6 in. or larger. The plate is securely fastened to the wall at the desired height and the base of the worker dropped in behind the metal strips to hold it firmly in position.

NOTE 3.—Bouncing the worker a few times on a table is very helpful in removing occluded air from soft lubricating greases.

The number of tests required shall conform to the requirements prescribed.

(c) A perforated cone has also been suggested,⁸⁷ which is allowed to fall under a given weight in a cylinder filled with the grease. The instrument is illustrated in Fig. 251, consisting of a base plate with a column and side arm to carry the split bearing; a water jacket to control temperatures; a cylinder to hold the sample; two interchangeable plungers; a set of weights; a "worker," consisting of a perforated disc at the end of a rod.

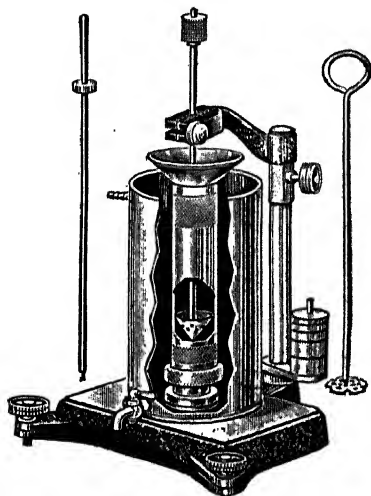
The characteristic features of the S.I.L. mobilometer are: (1) a perforated cone instead of a perforated plate descending through

the sample; (2) general design and construction with a view to maximum ease and convenience in operation.

The water jacket (bath) is of 1.9-liter (2-quart) capacity, with drain cock at bottom. It is permanently fastened to the base plate. The cylinder, which screws into the bottom of the water jacket, has a removable bottom to facilitate cleaning, and a filling mark

inscribed on the inside, 20 cm. from the bottom. Cylinders are interchangeable, so that several may be used with one instrument. Cylinder, with sample container, can be removed from the water jacket without draining the water.

The aluminum base plate, equipped with leveling screws and spirit level, supports a vertical rod and side arm which carries the split bearing directly above the center of the cylinder. The vertical support moves on a pivot, allowing the arm to swing away from the cylinder. The split bearing guides the plunger and permits the removal of the plunger assembly for cleaning before and after use.



Courtesy Precision Scientific Co., Chicago

FIG. 251.—S.I.L. Mobilometer for Testing Greases.

Interchangeable plungers weigh 15 g. and 90 g. respectively. Each has a weight platform at one end, and male thread at the other end for attachment of the perforated cone, weighing 10 g. Plungers are stainless steel, 30.5 cm. long, 6.35 mm. diameter, with two inscribed marks 10 cm. apart. Weights, ranging from 1 to 300 g., fit the plungers so that the load will be applied directly over the center.

(d) Another modification has been proposed for testing soft greases,⁸⁸ as follows:

Figure 252 shows a penetrometer assembled and equipped with the counterbalancing device, which is fixed to the back of the penetrometer by means of two screws that enter the holes provided for

securing the back plate of the original penetrometer. The modification is encompassed by *A*, a punctured screw to secure the thread; *B*, screws to secure the counterbalancing arm; and *C*, an aluminum cup to hold the counterbalancing weights. The two pulleys over which the thread passes are held between jeweled bearings. It can be installed or removed in a few minutes, although it does not inter-

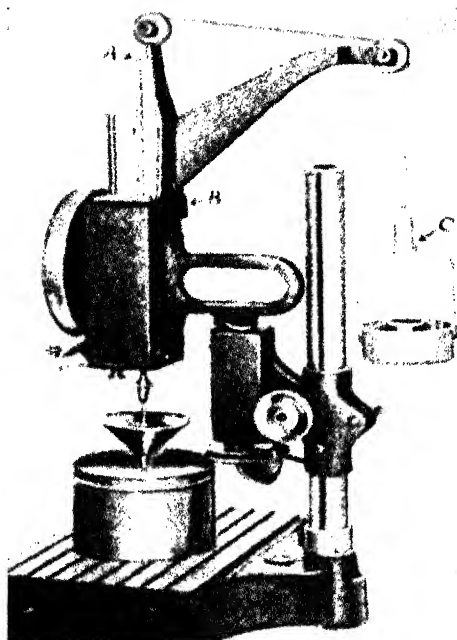


FIG. 252.—Modified Type of Penetrometer for Testing Soft Greases.

fere with regular A.S.T.M. penetrations when the thread is disconnected from the top of the shaft of the penetrometer.

In this manner the standard A.S.T.M. cone and attached moving parts are counterbalanced by means of a thread-arm-pulley-cup arrangement to reduce the force applied to the grease from the standard 150 g. to any desired lesser weight. It was found that 25 g. is a satisfactory minimum, less tending to give erratic results apparently due to unavoidable friction. Various types of fiber, horsehair, and catgut were tested in an effort to find a practical one with which friction will be low, and silk thread (Belding Hem-inway, pure silk A) was found most suitable.

(e) Additional methods have been proposed for testing the consistency of greases, including the measurement of the pressure required to force the substance through a capillary tube at a given temperature;⁸⁰ measuring the depth of penetration of a needle by means of a microscope;⁸¹ etc. Similarly, a procedure has been suggested for measuring the consistency of asphalt putties and asphalt-fibrous roof coatings, based on the principles of a falling cylindrical rod and steel ball, respectively;⁸² also a test measuring the extent of flow when subjected to pressure.⁸³

Test 9c. Consistometer. The principal shortcoming of the needle penetrometer is the fact that the readings at various temperatures (115, 77 and 32° F. respectively) must be expressed on *different* scales, and are therefore not comparable. It is difficult, and in many cases impossible, to interpret the *extent* of the physical change from the range in readings, upon subjecting a bituminous substance to variations in temperature. In addition, the scope of the penetrometer is limited, as it will not answer for either semi-liquid or hard bituminous materials. These objections are overcome in the consistometer. The consistometer is constructed according to scientific principles, and may be accurately duplicated at any time. It is suitable for all types of bituminous substances and registers the degrees hardness on a scale ranging from 0 to 100. It may be used for determining the hardness of substances as soft as vaseline (which will test 0.3 at 77° F.) to substances as hard as gilsonite (testing in the neighborhood of 100 at 77° F.). In all cases, the hardness or consistency is expressed as the cube root of the number of grams which must be applied to a circular flat surface 1 sq. cm. (100 sq. mm.) in area, to cause it to displace the substance at a speed of 1 cm. per minute. Readings for all bituminous substances and at all temperatures (whether 115, 77 or 32° F.) are expressed on a *single* scale. The harder the substance, the greater will be its hardness expressed numerically.

Four mushroom-shaped plungers are used, each having a round flat head with a reduced shank, so the perimeter of the penetrating surface forms a "knife" edge. This entirely eliminates the frictional adhesion of the bituminous substance to the sides of the plungers. The flat heads of the plungers are made in the following dimensions:

Plunger	Diameter in mm.	Area in sq. mm.
No. 1	1.13	1
No. 10	3.57	10
No. 100	11.28	100
No. 1000	35.67	1000

The method of testing consists in forcing one of the plungers into the substance at a *uniform* speed of 1 cm. per minute. The force is automatically registered in grams or kilograms. For any plastic substance the number of grams required to effect this displacement is directly proportional to the *volume* displaced. The volumes displaced per minute by the respective plungers are 0.01, 0.10, 1.00 and 10.0 ml. respectively. The relation between the plungers is therefore in the direct proportion of 1:10:100:1000.

For the sake of uniformity, all readings are expressed in terms of the number of grams applied to plunger No. 100 (1 sq. cm.). In other words, the readings obtained with plunger No. 1000 are divided by 10, those obtained with plunger No. 10 are multiplied by 10, and those obtained with plunger No. 1 are multiplied by 100. The hardness or consistency is equal to the cube root of this number of grams.

Two interchangeable springs are supplied, one registering in grams on a scale ranging 0 to 1000 g., in 10-g. divisions, and the

TABLE CXXI
RELATION BETWEEN CONSISTOMETER PLUNGERS

Plunger	Spring	Actual Reading	Converted to Grams per 100 sq. mm. Plunger	Cube Root Grams Applied 100 sq. mm. Plunger
1000 sq. mm.	G.	{ From 10 g. to 1000 g.	1 100	1.00 4.64
100 sq. mm.	{ G. Kg.	{ From 100 g. to 1000 g.	100 1,000	4.64 10.00
10 sq. mm.	Kg.	{ From 1.0 kg. to 10.0 kg.	1,000 10,000	10.0 21.5
1 sq. mm.	Kg.	{ From 1.0 kg. to 10.0 kg.	10,000 100,000 1,000,000	21.5 46.4 46.4 100.0

other for reading in kilograms on a scale ranging from 0 to 10 kgs., in 0.1-kg. divisions. In using plungers No. 1 and No. 10, the kilogram spring *only* should be employed; in using plunger No. 100 either the gram or the kilogram spring may be employed, depending upon the hardness of the material; in using plunger No. 1000, the gram spring *only* should be employed. The relations are expressed in Table CXXI.

Table CXXII shows the relation between the consistometer readings and degrees hardness, bearing in mind that in every case the hardness is designated as the cube root of the number of grams applied to the No. 100 plunger (area 100 sq. mm.), to cause it to displace the substance at a speed of 1 cm. per minute.

The consistometer is illustrated in Fig. 253. It is first levelled by the four screws *A*. The spring *B* is then attached, selecting the gram spring for soft substances, or the kilogram spring for hard substances. The steel shaft *C* is inserted and screwed firmly into place. The plunger *D* should then be screwed into the lower end of the shaft. Plunger No. 1 is used for hard and brittle substances, plunger No. 10 for moderately hard solid substances, plunger No. 100 for moderately soft semi-solid substances, and plunger No. 1000 for semi-liquid substances.

The scale *E* is graduated in grams on one side, and kilograms on the other, and is reversible. It should be inserted so that the graduations will correspond with the spring used, and adjusted so the indicator *F* will rest at the 0 division. The maximum indicator *G* is also brought to the 0 division, using the small fork *H*.

The bituminous substance is melted at the lowest possible temperature and poured into a small receptacle as described for the needle penetration method. The tin box *J* containing the bituminous substance is then supported underneath the machine in a vessel

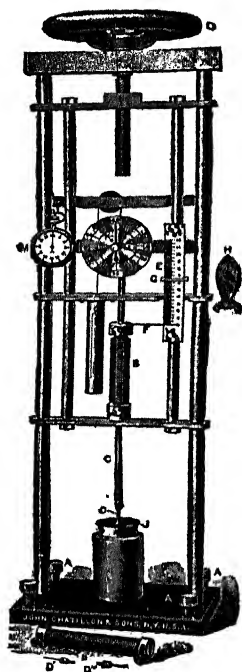


FIG. 253.—Consistometer.

TABLE CXXII
FOR CONVERTING CONSISTOMETER READINGS INTO DEGREES HARDNESS

PLUNGER NO. 1000 (1000 SQ. MM.) GRAM SPRING										
Grams Applied	0	10	20	30	40	50	60	70	80	90
0	0.00	1.00	1.26	1.44	1.59	1.71	1.82	1.91	2.00	2.08
100	2.15	2.22	2.29	2.35	2.41	2.47	2.52	2.57	2.62	2.67
200	2.74	2.76	2.80	2.84	2.88	2.92	2.96	3.00	3.04	3.07
300	3.11	3.14	3.17	3.21	3.24	3.27	3.30	3.33	3.36	3.39
400	3.44	3.45	3.48	3.50	3.53	3.56	3.58	3.61	3.63	3.66
500	3.68	3.71	3.74	3.76	3.78	3.80	3.83	3.85	3.87	3.89
600	3.91	3.94	3.96	3.98	4.00	4.02	4.04	4.06	4.08	4.10
700	4.12	4.14	4.16	4.18	4.20	4.22	4.24	4.25	4.27	4.29
800	4.31	4.33	4.34	4.36	4.38	4.40	4.41	4.43	4.45	4.46
900	4.48	4.50	4.51	4.53	4.55	4.56	4.58	4.59	4.61	4.63

PLUNGER NO. 100 (100 SQ. MM.) KILO SPRING										
Kilos Applied	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
100	4.64	4.79	4.93	5.07	5.19	5.31	5.43	5.54	5.65	5.75
200	5.85	5.94	6.04	6.13	6.21	6.30	6.38	6.46	6.54	6.62
300	6.69	6.77	6.84	6.91	6.98	7.05	7.11	7.18	7.24	7.31
400	7.37	7.44	7.50	7.55	7.61	7.66	7.72	7.77	7.83	7.88
500	7.94	7.99	8.04	8.09	8.14	8.19	8.24	8.29	8.34	8.39
600	8.43	8.48	8.53	8.57	8.62	8.66	8.71	8.75	8.79	8.84
700	8.88	8.92	8.96	9.00	9.04	9.09	9.13	9.17	9.21	9.24
800	9.28	9.32	9.36	9.40	9.44	9.47	9.51	9.55	9.58	9.62
900	9.65	9.69	9.73	9.76	9.80	9.84	9.86	9.90	9.93	9.97

PLUNGER NO. 10 (10 SQ. MM.) KILO SPRING										
Kilos Applied	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
1.0	10.0	10.5	10.6	10.9	11.2	11.3	11.7	11.9	12.2	12.4
2.0	12.6	13.8	14.0	14.2	14.4	14.6	15.75	14.9	14.1	14.3
3.0	14.4	14.6	14.7	14.9	15.0	15.2	15.3	15.5	15.6	15.7
4.0	15.0	16.0	16.1	16.3	16.4	16.5	16.6	16.75	16.9	17.0
5.0	17.1	17.2	17.3	17.4	17.5	17.65	17.8	17.9	18.0	18.1
6.0	18.2	18.3	18.4	18.5	18.6	18.7	18.8	18.85	18.95	19.0
7.0	19.1	19.2	19.3	19.4	19.5	19.6	19.7	19.75	19.8	19.9
8.0	20.0	20.1	20.2	20.25	20.3	20.4	20.5	20.6	20.65	20.7
9.0	20.8	20.9	20.95	21.0	21.1	21.2	21.25	21.3	21.4	21.5

PLUNGER NO. 1 (1 SQ. MM.) KILO SPRING										
Kilos Applied	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
1.0	21.1	21.2	21.3	21.4	21.5	21.7	21.8	21.9	22.2	22.7
2.0	27.1	27.6	28.0	28.4	28.8	29.2	29.6	30.0	30.4	30.7
3.0	31.1	31.4	31.7	32.1	32.4	32.7	33.0	33.3	33.6	33.9
4.0	34.2	34.5	34.8	35.0	35.3	35.6	35.8	36.1	36.3	36.6
5.0	36.8	37.1	37.3	37.6	37.8	38.0	38.3	38.5	38.7	38.9
6.0	39.1	39.4	39.6	39.8	40.0	40.2	40.4	40.6	40.8	41.0
7.0	41.2	41.4	41.6	41.8	42.0	42.2	42.4	42.5	42.7	42.9
8.0	43.1	43.3	43.4	43.6	43.8	44.0	44.1	44.3	44.5	44.6
9.0	44.8	45.0	45.1	45.3	45.5	45.6	45.8	45.9	46.1	46.3

PLUNGER NO. 1 (1 SQ. MM.) KILO SPRING										
Kilos Applied	0.0	0.1	0.2	0.3	0.4	0.5	0.6	0.7	0.8	0.9
1.0	10.1	10.2	10.3	10.4	10.5	10.6	10.7	10.8	10.9	11.0
2.0	18.5	18.6	18.7	18.8	18.9	19.0	19.1	19.2	19.3	19.4
3.0	26.9	27.0	27.1	27.2	27.3	27.4	27.5	27.6	27.7	27.8
4.0	35.3	35.4	35.5	35.6	35.7	35.8	35.9	36.0	36.1	36.2
5.0	43.7	43.8	43.9	44.0	44.1	44.2	44.3	44.4	44.5	44.6
6.0	52.1	52.2	52.3	52.4	52.5	52.6	52.7	52.8	52.9	53.0
7.0	60.5	60.6	60.7	60.8	60.9	61.0	61.1	61.2	61.3	61.4
8.0	68.9	69.0	69.1	69.2	69.3	69.4	69.5	69.6	69.7	69.8
9.0	77.3	77.4	77.5	77.6	77.7	77.8	77.9	78.0	78.1	78.2
10.0	85.7	85.8	85.9	86.0	86.1	86.2	86.3	86.4	86.5	86.6

of water (not shown) maintained at the temperature at which the test is to be performed. The pressure is applied to the plunger by turning the hand-wheel *O*, and the speed of displacement controlled by following the pointer *K* on the dial *L*, which should be caused to revolve at the same speed as the second hand of a chronometer *M*, conveniently suspended alongside. The numbers on the dial *L* correspond with those of the second hand on the chronometer. One revolution of the pointer *K* indicates that the plunger has moved downward exactly 1 cm.

At the termination of sixty seconds, after the pointer on the dial has made one revolution, the pressure on the plunger is relieved. The reading of the maximum indicator *G* on the scale *E* is then noted, and the corresponding degree of hardness ascertained by referring to the table.

When the plunger commences to displace the substance at the specified speed of 1 cm. per minute, a maximum reading is obtained which should remain constant throughout the entire displacement. The consistometer is simple to operate, gives closely concordant results, expresses the readings obtained at all temperatures on *one scale* and has a sufficiently great range to include all bituminous substances ordinarily encountered.⁹³

Test 9d. Susceptibility Index. Various methods have been suggested for evaluating the susceptibility of bituminous substances, so as to indicate the change of consistency or hardness with changes in temperature. It has been observed that substances having low susceptibility to temperature changes possess a high degree of plasticity, elasticity and thixotropy.⁹⁴

The following résumé is given of the various formulae that have been suggested from time to time for expressing the susceptibility of bituminous substances to changes in temperature:

1. Viscosity-temperature Indices (VT):

$$(a) \quad VT = \frac{\log V_1 - \log V_2}{\log t_2 - \log t_1}$$

where V_1 and V_2 are viscosity in poises at temperature t_1 and t_2 in °F., respectively. Low values indicate low temperature susceptibility.⁹⁵

$$(b) \quad VT = 100 [(V_1/V_2)^{1/(t_1-t_2)} - 1]$$

where V_1 and V_2 are absolute viscosity in poises at temperature t_1 and t_2 in °C., respectively. Low values indicate low temperature susceptibility.⁹⁶

$$(c) \quad VT = 0.221 \frac{\log \left[\frac{\log (V_1 + 0.8)}{\log (V_2 + 0.8)} \right]}{\log \left[\frac{t_2}{t_1} \right]}$$

where V_1 and V_2 are the kinematic viscosity in centistokes at two standardized temperatures t_1 and t_2 (e.g., 210° F. and 275° F.), respectively. Low values indicate low temperature susceptibility.⁹⁷

2. Penetration-temperature Indices (PT):

$$(a) \quad PT = \frac{\text{Penetration at } 46.1^\circ \text{ C. per 50 g. per 5 sec.} - \text{Penetration at } 0^\circ \text{ C. per 200 g. per 60 sec.}}{\text{Penetration at } 25^\circ \text{ C. per 100 g. per 5 sec.}}$$

$$(b) \quad PT = \frac{\text{Penetration at } 37.8^\circ \text{ C. per 100 g. per 5 sec.}}{\text{Penetration at } 25^\circ \text{ C. per 100 g. per 5 sec.}}$$

$$(c) \quad PT = \frac{\text{Penetration at } 25^\circ \text{ C. per 100 g. per 5 sec.}}{\text{Penetration at } 0^\circ \text{ C. per 200 g. per 60 sec.}}$$

Low values indicate low temperature susceptibility.⁹⁸

$$(d) \quad PT = \int_{t_1}^{t_2} dp/dt$$

where $dt = p_1 - p_2$, and p_1 and p_2 are the penetrations at t_1 and t_2 , respectively (using the same weight and time interval). Low values indicate low susceptibility.⁹⁹

3. Viscosity-penetration-temperature Indices (VPPT):

$$(a) \quad VPPT = 1/100 (\text{Saybolt furol visc. at } 275^\circ \text{ F.} - \text{Pen. at } 77^\circ \text{ F., 100 g., 5 sec.}) (\text{Pen at } 77^\circ \text{ F., 100 g., 5 sec.})$$

High values indicate low temperature susceptibility.¹⁰⁰

$$(b) \quad \eta = \frac{5.9 \times 10^9}{(\text{Penetration})^{1.93}}$$

where η = viscosity in poises, and p = penetration expressed in decimillimeters. This is known as the "Saal formula," and is suitable for penetrations above 55 decimillimeters. Low values indicate low temperature susceptibility.¹⁰¹

4. Softening Point-penetration-temperature Indices (SPT):

$$(a) \quad SPT = \left(\frac{\text{Consistometer Hld. at } 32^\circ \text{ F.} - \text{Consistometer Hld. at } 115^\circ \text{ F.}}{\text{K. and S. Fusing-point } ^\circ \text{ F.}} \right) \times 100$$

Low values indicate low temperature susceptibility. By means of this particular index, bituminous materials may be roughly divided into the following groups,¹⁰² viz.:

Index under 40: Includes blown petroleum asphalts, fatty-acid pitches and fluxed asphaltites (having an index between 8 and 40); also wurtzilite asphalts (having an index between 30 and 40).

Index between 40 and 60: Includes residual asphalts.

Index over 60: Includes mineral waxes, pitches derived from tars, and asphaltites (of which the susceptibility index varies from 75 to over 100).

Native asphalts have been excluded from the foregoing groups, since their susceptibility indices vary widely, ranging from 15 to greater than 100. The author has never examined a bituminous material having a susceptibility index lower than 8.

$$(b) \quad SPT = \frac{\log 800 - \log \text{pen. at } 77^\circ \text{ F., } 100 \text{ g., } 5 \text{ sec.}}{\text{R. and B. Fusing-point } ^\circ \text{ F.} - 77}$$

This formula is based upon the assumption that the penetration of all asphalts at their softening point is approximately 800 (which has not thus far been refuted). Low values indicate low temperature susceptibility.¹⁰³

DUCTILITY

This represents the capacity of the bituminous material for elongating or stretching. The test is of value for identifying the bituminous substance, for indicating its adaptability in connection with certain usages, for gauging its uniformity of supply, and for

purposes of factory control. The ductility test will often differentiate blown petroleum asphalts from native or residual asphalts. Most pitches derived from tars are extremely ductile, but fatty-acid pitches are variable in this respect. The ductility test is useful for predetermining the adaptability of bituminous materials for paving purposes, for adhesive compounds to be used in connection with waterproofing or built-up roofing work, and for manufacturing surface coatings of preparing roofings. Wherever the bituminous material is subjected to extensive changes in temperature or vibration, it is important that it should have high ductility within the particular temperature range to which it will be subjected. With every bituminous substance there exists a certain temperature, usually within 10 to 30° F. of its fusing-point (K. and S. method), at which the ductility attains a maximum. A ductility curve constructed for any bituminous substance over a range of temperature assumes the same form as the probability curve in higher mathematics. It is desirable that the maximum ductility should coincide as closely as possible with the *average* temperature to which the material is to be subjected during use.

There are two methods in use, depending upon the construction of the molds, namely one devised by A. W. Dow,¹⁰⁴ and one proposed by the author.

Test 10a. Dow's Method. This test has been standardized as follows:¹⁰⁵

The ductility of a bituminous material is measured by the distance to which it will elongate before breaking when two ends of a briquette of the material of the form described are pulled apart at a specified speed and at a specified temperature. Unless otherwise specified, the test shall be made at a temperature of 25° C. \pm 0.5° C. (77° F. \pm 0.9° F.) and with a speed of 5 cm. per minute (\pm 5.0 per cent).

The mold shall be similar in design to that shown in Fig. 254. Dimensions shall be as given with the permissible variations indicated. The mold shall be made of brass, the ends, *b* and *b'*, being known as clips, and the parts, *a* and *a'*, as sides of the mold. The dimensions of the mold shall be such that, when properly assembled, it will form a briquette having the following dimensions:

Total length.....	7.45 to 7.55 cm.
Distance between clips.....	2.97 to 3.03 cm.
Width at mouth of clip.....	1.98 to 2.02 cm.
Width at minimum cross-section (halfway between clips)....	0.99 to 1.01 cm.
Thickness throughout.....	0.99 to 1.01 cm.

The water bath shall be maintained at the specified test temperature varying not more than 0.1°C . (0.18°F .) from this temperature. The volume of water shall be not less than 10 liters and the sample shall be immersed to a depth of not less than 10 cm.

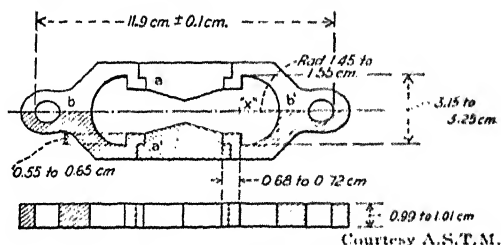


FIG. 254.—Ductility Mold.

NOTE.—The opening in the end of each clip, as indicated by "x," shall be half an ellipse having a transverse axis of 3.2 cm. \pm 0.05 cm. and half of the longitudinal axis shall be 1.45 to 1.55 cm.

and shall be supported on a perforated shelf not less than 5 cm. from the bottom of the bath.

For pulling the briquette of bituminous material apart, any apparatus may be used that is so constructed that the briquette will be continuously immersed in water as specified, while the two clips are pulled apart at a uniform speed, as specified, without undue vibration.

The bituminous material to be tested shall be completely melted until thoroughly fluid by heating it in an oil bath maintained at the minimum temperature needed to properly liquefy the sample (see Note). It shall then be strained through a No. 50 sieve and, after a thorough stirring, poured into the mold. The mold shall be assembled on a brass plate and, so as to prevent the material under test from sticking, the surface of the plate and interior surfaces of the sides *aa'* of the mold shall be thoroughly amalgamated.* The

* The amalgamation may best be effected by immersing the clean mold in a solution of mercury bisulfate containing free metallic mercury, and so as to come in contact with the latter. Instead of mercury, the metal mold, preferably of stainless steel, may be moistened with glycerol.¹⁰⁸

plate upon which the mold is placed shall be perfectly flat and level so that the bottom surface of the mold will touch it throughout.¹⁰⁷ In filling the mold, care shall be taken not to disarrange the parts and thus distort the briquette. In filling, the material shall be poured in a thin stream back and forth from end to end of the mold until it is more than level full. It shall be left to cool to room temperature and then placed in the water bath maintained at the specified temperature of test for a period of 30 to 40 min., after which the excess bitumen shall be cut off by means of a hot straight-edged putty knife or spatula so that the mold shall be just level full.

NOTE.—When paving asphalt cements are being tested, the oil bath shall be maintained at a temperature of from 150 to 160° C. (302 to 320° F.).

The brass plate and mold, with briquette, shall then be placed in the water bath and kept at the specified temperature for a period of from 85 to 95 min., when the briquette shall be removed from the plate, the side pieces detached, and the briquette immediately tested. The rings at each end of the clips shall be attached to the pins or hooks in the ductility machine and the two clips pulled apart at a uniform speed as specified¹⁰⁸ until the briquette ruptures. A variation of ± 5 per cent from the speed specified will be allowed. The distance through which the clips have been pulled to produce rupture shall then be measured in centimeters. While the test is being made, the water in the tank of the ductility machine shall cover the sample both above and below it by at least 2.5 cm. and shall be kept continuously at the temperature specified within $\pm 0.5^{\circ}$ C. ($\pm 0.9^{\circ}$ F.).

A normal test is one in which the material between the two clips pulls out to a point or thread until rupture occurs at the point where the thread has practically no cross-sectional area. The average of three normal tests shall be taken and reported as the ductility of the sample.

If the bituminous material comes in contact with the surface of the water or the bottom of the bath, the test shall not be considered normal.

NOTE.—When the specific gravity of the bituminous material to be tested is below 0.98 or above 1.02, the specific gravity of the water bath in the ductility machine shall be made the same as the material to be tested by the addition of either methyl alcohol or sodium chloride.

If a normal test is not obtainable on three successive tests, the ductility shall be reported as being unobtainable under the conditions of the test.

It is customary to find the ductility at three temperatures, viz.: 115°, 77° and 32° F. However, considerable insight may be ob-

TABLE CXXIII
DUCTILITY AT VARYING TEMPERATURES

Temp.	A	B	C	D	E	F	G	H
-3° C.	7.7	7.1
-2°	8.5	9.4
0°	0	12	10.5
+1°	1.5	0
1.5°	0	2.0
2°	2	0	30.5	5	16	14.5
2.5°	10.5
3°	0	8.2	61	32.5
3.5°	1
4°	>100	14	8	94	64	25	25
5°	0	33	22	>100	>100	30.5
6°	1	61.5	32	34
6.5°	>100
7°	81	41	46
7.5°	69
8°	>100
9°	98	52
11°	65	79.5
13°	80.5	>100
15°	>100

Where *A* represents soft coal-tar pitch.

B the same distilled to a fusing-point of 28° C. (loss 8.4 per cent).

C a mixture of coal-tar pitch and anthracene oil (70/30).

D the same as *C* distilled to a fusing-point of 28° C. (loss 9.0 per cent)

E a mixture of coal-tar pitch and heavy oil (75/25).

F the same as *E* distilled to a fusing-point of 28° C. (loss 9.5 per cent).

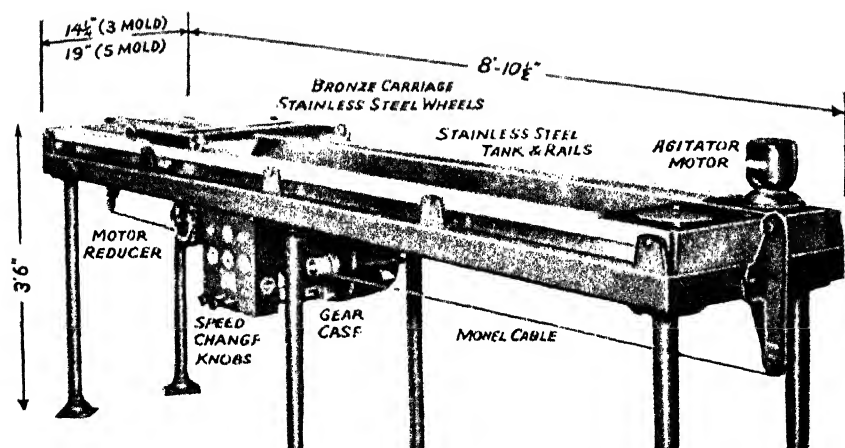
G and *H* soft residual asphalts having fusing-points of 28° C.

tained regarding the origin and characteristics of bituminous substances, by ascertaining the ductility over an extended temperature range, as may be noted from the figures in Table CXXIII.¹⁰⁹

Various machines have been devised for ascertaining the ductility, in which the molds are drawn apart mechanically, including the one illustrated in Fig. 255.¹¹⁰

This improved model ductility machine consists of a stainless steel double wall tank, provided with a circulating liquid between

the walls. The liquid is preferably water which is maintained automatically at a constant temperature of 77°F . by means of a cooling coil and an electric heating element actuated by a sensitive thermoregulator. The circulating mechanism is compact and consists of a motor-driven centrifugal pump, arranged so that, by baffling the jacketed cooling medium, a positive circulation of that medium is insured. By setting the thermoregulator for the desired



Courtesy Emil Greiner Co., New York

FIG. 255.—Ductility Machine.

temperature, the water in the jacket is maintained at the proper temperature and the bath medium in which the actual test is made is thereby also held at this constant temperature without agitation.

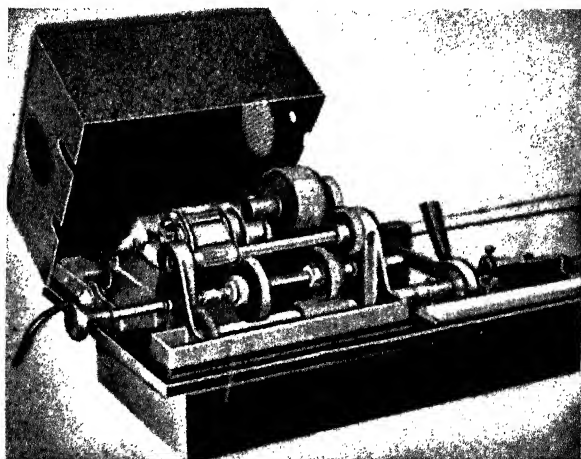
The machine has been designed in two sizes: one model will accommodate 3 test specimens and a larger model is for 5 test specimens.

The driving mechanism shown in Fig. 256 provides for various speeds, any one of which is occasionally required by purchasing specifications for asphalt, viz.: 0.25, 1, 5 cm. There is also available a 20 cm. per minute speed, which can be obtained in either the 3-mold machine or the 5-mold instrument, if required.

The gear change housing is directly connected to a gear head motor and all speed changes can be made by two control knobs on

the panel. There is also provided a neutral, whereby the power unit may be left running while the carriage is idle.

The usual centimeter scale is provided for reading distance to which the specimens are pulled out before breaking.



Courtesy Precision Scientific Co., Chicago

FIG. 256.—Gear-shift Speed Control for Ductility Machine.

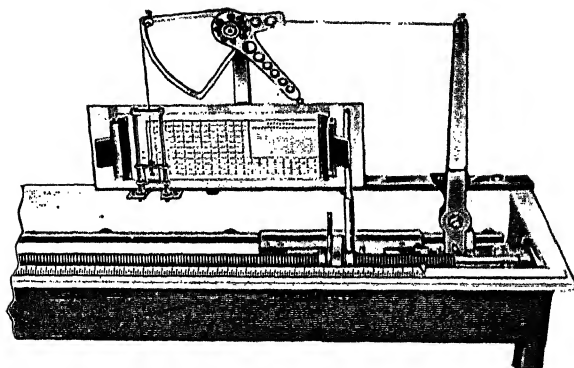


FIG. 257.—Ductility Tension Machine (Humboldt Type).

Another type of ductility tester, illustrated in Fig. 257, is designed to measure strength tests in addition to the ductility test. The load required to pull the specimen is transmitted to the weighing device and the weight is recorded on a chart, which is fastened to the moving carriage of the machine, and runs through a guide at the rear of the recording pen.

An instrument with a dynamometer attachment adapted to use the Dow mold, measuring both the ductility and "cementitiousness" (tensile strength) has been described by Lester Kirschbraun.¹¹¹ This device is essentially the same as that which had been previously described by the author. (See Test 10*b*.)

It has been shown that partially blown asphalts may be prepared which will show a ductility at 32° F. of at least 1 cm. by the above method of testing, some samples running as high as 2.5 cm.¹¹²

Test 10*b*. Author's Method. An improved mold designed by the author is illustrated in Fig. 258 and shown in cross-section in Fig. 259. It consists of two cylindrical sections constructed of hard-

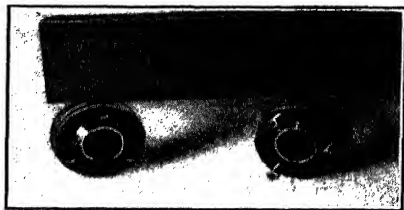


FIG. 258.—Author's Ductility Mold.

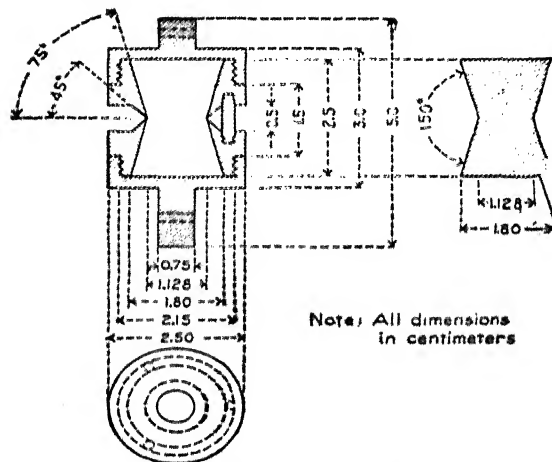


FIG. 259.—Cross-section, Author's Mold.

ened steel, resting together on circular knife-edges and maintained in that position by three guide pins. It is filled by unscrewing the upper cap and pouring in the melted bituminous substance, which on cooling forms a prismoid, whose altitude is 2.5 cm., the end-areas 1.8 cm. in diameter, with a minimum cross-section at the center of exactly 1.0 sq. cm. (1.28 cm. in diameter). The upper cap is screwed in place, the mold fastened in the tensometer and the two halves separated at the uniform speed of 5 cm. per minute. The elongation in cms. at the moment the material parts is a measure of its ductility.¹¹³

The mold has a number of advantages over the Dow type, including its adaptability to testing semi-liquid and semi-solid bituminous materials, no amalgamation is necessary, there is no danger of the material breaking in the mold upon

being cooled to the proper temperature, the personal equation is eliminated in filling the mold with the assurance that the minimum cross-section will be *exactly* the proper size, and only a small quantity of the material is required in making the test.

The tensometer is illustrated in Fig. 260. The two sections of the mold *A-1* and *A-2* are clamped between the guides *B-1* and *B-2*, the lower section being

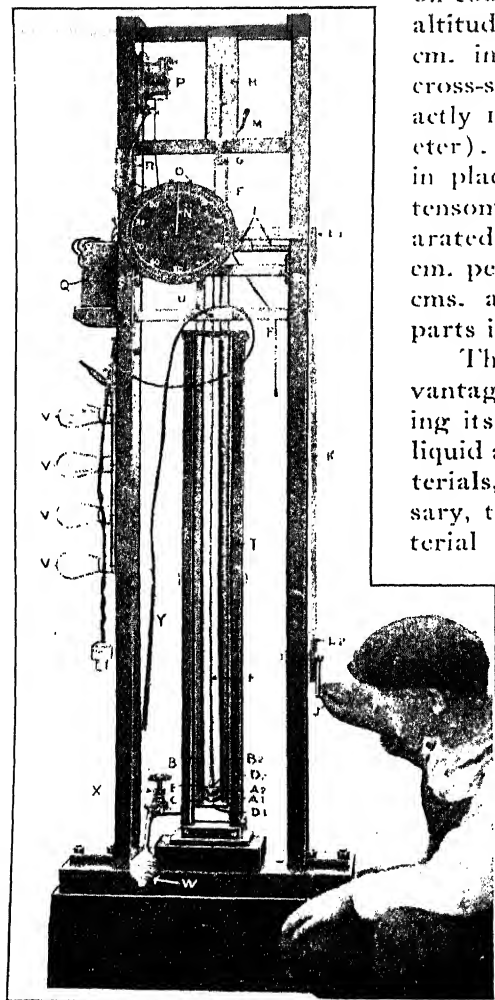


FIG. 260.—Tensometer.

fastened to the stationary cross-piece *C* by the pin *D*-1, and the upper section to the movable cross-head *E* by the pin *D*-2. The cross-head is attached to the chain *F* which passes over the sprocket-wheel *G* fastened to the dynamometer *H*, and then around a suitable winding mechanism *I*. The specimen is drawn apart by turning the handwheel *J*, which operates the endless chain *K* running on the sprocket wheels *L*-1 and *L*-2. The dynamometer is equipped with a trigger *M* to prevent recoil. The chain *F* also connects with a train of gears operating the brass pointer *N* pressing against the dial *O* which is formed of vulcanite or some other insulating material. Its face is marked in one hundred divisions, each consisting of a metallic contact. As the pointer brushes over these contacts it momentarily closes an electric circuit which operates the relay *P*, causing a "click." The relay is connected with the batteries *Q* and the switch *R*. One revolution of the pointer indicates that the halves of the mold have been separated a distance of exactly one meter, and a movement of the pointer over one division of the dial corresponds to a centimeter rise in the section *A*-2.

The guides *B*-1 and *B*-2 are pivoted at *S*-1 and *S*-2, which permits the glass reservoir *T* being slipped into place, whereupon they are locked into position by the bolt *U*. The reservoir is filled with water maintained at the desired temperature by the heating coil *Y* in series with the incandescent lamps *V*. The bath may be agitated upon squeezing the bulb *W*, which forces air through the liquid. The valve *X* is for emptying the reservoir. The speed is controlled by a metronome with a bell attachment set to ring every 12 seconds, or 5 times per minute. The speed with which the crank is turned must be regulated so that the "clicks" of the relay are brought into unison with the rings of the metronome.

The reservoir should be filled with a liquid having about the same specific gravity as the bituminous material tested, so the thread of material will neither have tendency to float nor sink while the molds are being separated. The operator must watch the specimen as sections *A* and *B* separate, and he should cease turning the crank at the moment the thread parts. The dynamometer indicates the tensile strength of the substance in kilograms (Test 11), and the dial *O* records its ductility in centimeters. The substance is usually tested at 115, 77 and 32° F.

TENSILE STRENGTH (COHESIVENESS)

Cohesion (cohesiveness) is defined as the force by which molecules of the same substance are held together, and causes the substance to resist being pulled apart. The cohesion of bituminous substances is a function of the hardness (penetration). The softer the substance (derived from the same source), the less will be its cohesiveness. The cohesion of a liquid at a given temperature has been shown to be equal to twice its surface tension at the same temperature. This may be explained by the fact that molecules of a liquid are surrounded by a field of force, and when the molecules are in the body of the liquid, this attraction for its neighbors is the same in all directions. However, the molecules at the boundary between the liquid and air are attracted to one another laterally on the surface only, creating a tension, known as the surface-tension.

Test 11. Author's Method. The tensile strength (cohesiveness) of bituminous substances may be measured on the tensometer as described in Test 10b, and is equal to the maximum reading in kilograms as the two halves of the mold separate. It is a measure of cohesiveness or cementitiousness and is of value in determining the adaptability of a bituminous substance for certain definite purposes, especially for paving, manufacturing adhesive compounds for waterproofing and built-up roofing work, bituminous substances for electrical insulation, molded articles, pipe joints, etc. The tensile strength is ordinarily tested at 115, 77 and 32° F. For each bituminous substance, there is a critical temperature at which the tensile strength reaches a maximum, and this is generally coincident with the temperature at which the ductility approaches 0. This phenomenon may be explained by the disappearance of plasticity and associated cohesiveness at temperatures when the substance becomes transformed into a brittle solid. The tensile strength curve is also similar in form to the probability curve in higher mathematics. There appears to be no definite relation between the hardness and tensile strength of bituminous substances. With residual asphalts manufactured from the same crude, the tensile strength is reduced after the distillation progresses beyond the hard and brittle stage. Excessive blowing produces the same results, but to a lesser degree.

ADHESIVENESS

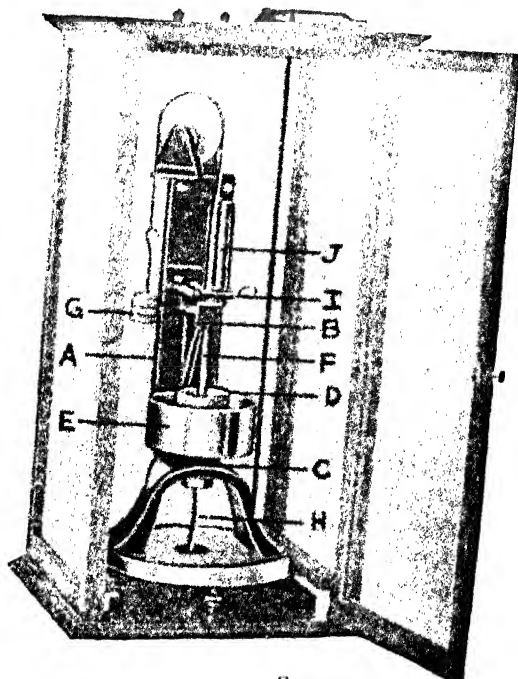
Adhesion (adhesiveness) is defined as the force by which one substance adheres to another of a different nature.¹¹⁴ This test serves as a measure of the adhesiveness of the bituminous material, and it is of primary importance in ascertaining its adaptability for certain definite usages, as, for example, in road building, preparing compounds for waterproofing and built-up roofing work, cements, etc. It represents the capacity of the substance to adhere to solid objects with which it may be brought in contact, and differs entirely from the cohesiveness or tensile strength referred to in Test 11. Various instruments have been proposed for this purpose, including those devised by Fulweiler,¹¹⁵ Osborne,¹¹⁶ Brown, Kirschbraun and others.¹¹⁷

Test 12a. Riehm's Method. This test constitutes a rough indication of the adhesiveness of the substance and is ascertained by noting the number of seconds time it takes for a 10-gram weight (the underside of which is smooth and clean) to adhere to a freshly prepared surface of the bituminous substance at 20° C.¹¹⁸

Test 12b. Wedmore's Method. A cylindrical steel mold, open at both ends, is allowed to rest against the surface of a polished steel plate, and filled with the melted bituminous substance at a predetermined temperature. Upon cooling to 77° F., the mold is subjected to a steady, slow pull, and the force measured which is necessary to separate the mold from the plate. The area of adhesion should be \leq 90 per cent of the whole area covered by the specimen.¹¹⁹

Test 12c. Brown's Method. Performed by means of an instrument designed by W. A. Brown, termed the "adhesivimeter," for measuring the stickiness or cohesive strength of road oils. The instrument is illustrated in Fig. 261 and consists of a cast-iron frame *A* carrying two brass bearings. The upper bearing *B* is 2 in. long and the lower bearing *C* is $\frac{1}{2}$ in. in diameter and $\frac{1}{2}$ in. long, attached to an oil-cup *D* which in turn is surrounded by the water bath *E*. A steel rod *F* passes through the bearings and is counter-balanced by the weight *G*. The testing weights are suspended from the wire *H* attached to the lower end of the rod. A clamp *I* holds

the rod in position prior to the test. In running the test, the oil-cup *D* is filled with the road oil which serves to coat the rod before it reaches the lower bearing *C*. When the oil has reached the desired temperature, controlled by the water bath provided with an



Courtesy of Braun Corp.

FIG. 261.—Brown Adhesivimeter.

electric heater and thermostat, the rod is raised until its upper end reaches the zero mark on the scale *J*. The clamp is then released, and when the upper end of the rod has fallen to the 3-cm. mark, a stop-watch is started and timed until it reaches the 13-cm. mark. The time in seconds for the rod to fall 10 cm. is an index of the adhesiveness of the material. The test is usually run at 95° F. with a weight of 760 g. suspended from the lower end of the rod.

SURFACE TENSION

Test 12d. Nellensteyn's Method. A special form of apparatus has been devised by F. J. Nellensteyn¹²⁰ for ascertaining the surface-tension of bituminous substances, as shown in Fig. 262, consisting of a manometer which is formed of two hollow cylinders *A* and *B* joined together by an air-tight closed coupling *C*. The cylinders are partly filled with dekalin (*D*) which supports a duraluminum

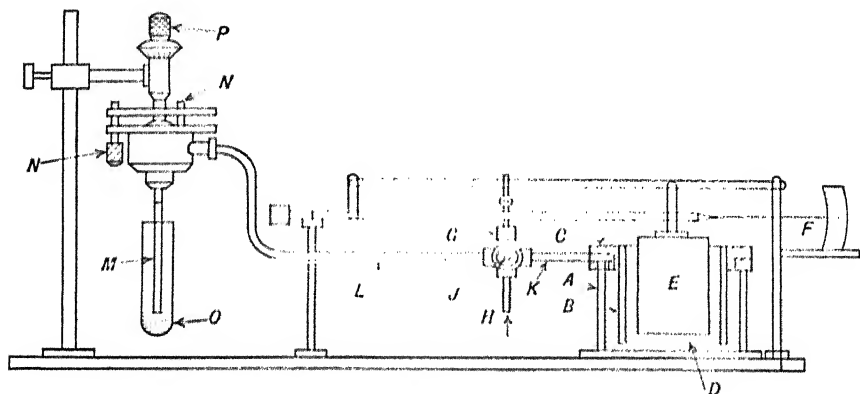


FIG. 262.—Apparatus for Determining Surface-tension.

float *E* of accurate dimensions. This float is attached to a weighing device. As the pressure in the gas-chamber of the manometer increases, the pointer *F* moves upwards and can be recorded by the weight *G*. Purified nitrogen gas (washed with alkaline pyrogallol, followed by alkaline manganese chloride solution) enters at *H* and the pressure is regulated by the needle-valve *J*, which is connected with the manometer by the tube *K*, and with the capillary *M* by the tube *L*. The capillary is composed of a platinum-rhodium alloy and has a diameter at the tip of 0.6 mm. It is adjusted in a vertical position by the set-screws *N*, whereupon the tip of the capillary is introduced just below (i.e., 0.1 mm.) the level of the liquid to be tested *O*, by a micrometer screw *P*. The bituminous substance is maintained at the desired test-temperature by being surrounded by a bath of castor oil (not shown).

The apparatus is first calibrated against C.P. benzol, which has

a surface-tension of 28.5 dynes/cm. at 20° C., with a temperature correction of 0.16 dyne/cm. per degree C. Upon making the test, the zero-point is obtained by allowing the gas to enter under the greatest pressure which will not, however, cause it to bubble through the substance under test. The pressure is then gradually increased until the gas bubbles through the substance at the rate of 1 bubble per minute, and thereupon gradually reduced until a point is reached where there is no noticeable further change in pressure. This ordinarily takes place when the bubbles form at the speed of one in each 30 to 50 seconds, before they burst. This pressure is then recorded.

The surface-tension may be calculated by means of Cantor's equation, which in simplified form, gives the following:

$$\alpha = \frac{rH}{2}$$

where α = surface-tension energy expressed in ergs per cm.²,

r = radius of the tip of capillary tube in mm.,

H = pressure recorded in dynes (i.e., mm. mercury corrected to 0° C. \times 1333.2 dynes).

The molecular surface-tension is calculated from the following formula:

$$\mu = \alpha \left(\frac{M}{d} \right)^{\frac{2}{3}}$$

where μ = molecular surface-tension energy in ergs per cm.²,

M = molecular weight of the substance,

d = specific gravity of the substance at the temperature of test, compared with water at 4° C.

Nellensteyn reports the figures given in Table CXXIV, upon examining the following substances:

A. Blown Venezuelan petroleum asphalt (R. and B. fusing-point 64.2° C.).

B. Residual Mexican petroleum asphalt (R. and B. fusing-point 42° C.).

C. Residual Mexican petroleum asphalt (R. and B. fusing-point 55.7° C.).

D. Residual Mexican petroleum asphalt (R. and B. fusing-point 58.5° C.).

E. Trinidad asphalt separated from its associated mineral constituents by dissolving in CS_2 and centrifuging at 40,000 r.p.m. (R. and B. fusing-point $65^\circ \text{C}.$).

F. Coal-tar pitch obtained by distilling coal tar to $300^\circ \text{C}.$ (R. and B. fusing-point $60.2^\circ \text{C}.$, free carbon 10.7 per cent).

These investigations show that whereas all true liquids exhibit a changing surface-tension proportional to the temperature of test, both asphalts and pitches show a steadily falling surface-tension to a certain value, as the temperature rises, at which point the curve suddenly flattens, after which the surface-tension again becomes proportional to the temperature, the fall thereupon being more gradual than previously. This indicates a change of state at this critical point, which has a different value for the different substances examined.

TABLE CXXIV
SURFACE-TENSION OF ASPHALTS AND THEIR COMPONENTS

	A		B		C		D		E		F	
	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.	Temp. °C.	S.T. dynes per cm.
<i>Surface Tension (S.T.) Original Substance:</i>												
Range examined	117	33.4	93.5	31.9	117	31.9	116	32.7	108	37.4	67	39.7
Bend in S.T.-Temp. chart occurs at	225	20.7	179	23.8	224	22.1	215	22.3	193.5	25.8	180	33.8
$d(\text{S.T.}) \div d(\text{Temp.})^*$ from bend in chart to highest temperature examined	130	24.8	117	27.5	145	26.7	138	26.8	131.5	30.1	80	38.8
	0.0546		0.5095		0.0561		0.0584		0.0603		0.0530	
<i>Surface Tension (S.T.) Separated Oily Constituents (Note A):</i>												
Range examined	30	36.6	39.5	34.7	52	35.7	45	33.5	65	34.8	46	46.5
Bend in S.T.-Temp. chart occurs at	226	20.5	184.5	22.8	233	21.3	220	21.5	128	20.9	211	30.8
$d(\text{S.T.}) \div d(\text{Temp.})^*$ from bend in chart to highest temperature examined	50	30.6	58	30.6	110	28.2	69.5	30.3	80	30.0	65	38.6
	0.0575		0.0600		0.0553		0.0584		0.0615		0.0522	
<i>Relation Between S.T., Original Substance and Separated Oily Constituents (Note A):</i>												
Difference between S.T.s at 180° C. of original substance and separated oily constituents	—	0.25	—	0.60	—	0.35	—	0.50	—	2.85	—	1.25
Difference between temperatures of Bends in S.T.-Temp. charts of original substances and separated Oily Constituents	100	—	65	—	85	—	70	—	55	—	15	—

* The temperature coefficient of surface tension per $^\circ \text{C}.$

NOTE A.—The separated oily constituents (i.e., the "medium") were isolated by dissolving in petroleum naphtha, allowing to stand, chilling and then filtering. Acetone was then added to the extract, allowed to stand filtered and the extract evaporated to dryness.

Of all the substances tested, Trinidad asphalt showed the highest $d(\text{S.T.})/d \text{ Temp.}$ value. This undoubtedly accounts for its greater miscibility with coal-tar products than proves to be the case with petroleum asphalts—even the softer varieties.

The oily constituents show a lower surface-tension than the original substance, at any given temperature. The greatest difference is noted in the case of Trinidad asphalt; coal-tar pitch comes next; and petroleum asphalts come last.

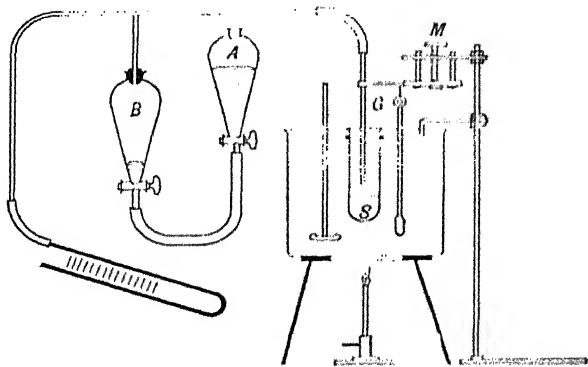


FIG. 263.—Modified Apparatus for Surface-tension.

Nellensteyn's bubble method has been found most satisfactory for testing tars between 80 and 90° C., whereas the Du Noüy instrument with a platinum ring of 4-cm. periphery gives the most satisfactory results at room temperatures. Nellensteyn's observations when plotted at different temperatures show a sudden break in the surface-tension readings at lower temperatures, due no doubt to the interfering effect of high viscosities. The method may be simplified as follows:¹²¹

The tar is brought into sample tube *S* and immersed in a constant-temperature bath (Fig. 263). After the desired temperature equilibrium has been reached, the glass tube, *G*, provided with a fine opening at the end, is gently lowered until its tip touches the surface of the tar. Contact is readily obtained by use of the screw arrangement, *M*. Then, the two stopcocks are adjusted so that the mercury in container *A* flows slowly into container *B*. Since the air in

B is gradually displaced by mercury, pressure is developed in the system. At a definite maximum pressure the air bubble formed on the tip of the glass tube bursts, thereby releasing part of the pressure in the system. This maximum pressure is a direct measure of the surface tension of the liquid under consideration.

Since an inclined gage is used, an over-all constant for the apparatus should be ascertained by measuring the maximum bubble pressure for water, nitrobenzene and benzene. In the case of tars, it is advisable to reduce the air displacement to not exceeding two bubbles per minute.

Test 12c. Interfacial Tension. Interfacial tension between bituminous materials and aqueous media is an important consideration in studying dispersions. Various methods have been suggested, but the most accurate has been devised by P. L. Du Noüy,¹²² known as the "tensiometer." The procedure is as follows:

The bath used to maintain the temperature for the measurements with the tensiometer, consists of a large crystallizing dish held in place on a plate 18 cm. in diameter by upright arms of spring steel. The plate is fastened to the table of the tensiometer by means of bolts. The dish is filled to about three-fourths capacity with Nujol and equipped with an electrical heating element, thermometer, and mechanical stirrer. An 8-cm. Pyrex crystallizing dish, in which were placed the water or aqueous solution and the asphalt to be investigated, is held in a rigid clamp which fitted to the side and has legs resting on the bottom of the bath. The temperature of the bath is maintained at approximately 85° C. A change of 2° or 3° C. in the temperature has no measurable effect on the values of the interfacial tension.

Since the asphaltic materials have a density slightly lower than that of the water or solution, the latter is placed in the small crystallizing dish, and the platform of the tensiometer raised until the ring is immersed. The asphalt heated to about 85° C. is then carefully poured on the surface of the aqueous phase. After the materials have reached the desired temperature, the ring is pulled slowly upward against the interface, and the platform lowered to keep the pointer on the index line. Three to four minutes are consumed in changing the reading on the dial by one dyne. When the ring breaks through the interface, the reading on the dial is recorded. The instrument is calibrated frequently to indicate the interfacial tension directly in dynes per centimeter.

Two different types of asphaltic materials were investigated.

They are distinguished by the letters *A* and *B*. Flux *A* was derived from a Venezuelan asphaltic petroleum by steam distillation, and flux *B* from a Trinidad asphaltic petroleum by the same process; their physical properties being as follows:

	Flux A	Flux B
Sp. gr. at 85/85° C.	0.9964	0.9970
Float at 150° F. (65.6° C.), seconds.	44	53
Flash (Cleveland open cup), " F. (" C.)	420 (215.6)	415 (212.8)

The values obtained using fluxes *A* and *B* and sodium hydroxide solutions from 0.0013 to 0.25 *M* are as follows:

NaOH Concn., Mole/liter	Interfacial Tension		NaOH Concn., Mole/liter	Interfacial Tension	
	Flux A, Dynes/cm.	Flux B, Dynes/cm.		Flux A, Dynes/cm.	Flux B, Dynes/cm.
0.0000	16.7	15.7	0.0100	0.5	0.0*
0.0013	11.4	7.9	0.0500	0.0*	0.0*
0.0025	9.5	5.3	0.1000	0.0*	0.0*
0.0050	5.3	0.0	0.2500	0.0*	0.0*

* Emulsification apparently took place at the interface. The aqueous solution contained dispersed material which in time made the solution opaque.

These data indicate that flux *B* contained material more reactive toward sodium hydroxide than flux *A*, the resulting compounds causing the lowering of the interfacial tension.

(C) THERMAL TESTS

THERMAL CONDUCTIVITY

The following symbols and equations have been proposed¹²³ for designating heat transmission:

Area	<i>A</i>
Temperature, deg. Fahr. or deg. Cent.	<i>t</i>
Temperature, deg. Fahr. absolute or deg. K (deg. Cent. absolute)	<i>T</i>
Length of path of heat flow (thickness)	<i>L</i>
Total quantity of heat transferred	<i>Q</i>
Time (when <i>t</i> is used for temperature)	<i>τ</i>
Thermal transmission (heat transferred per unit time)	<i>q</i>

$$- - Q$$

Thermal conductivity (heat transferred per unit time per unit area, and per degree per unit length)

$$k = \frac{q}{\frac{A}{(t_1 - t_2)}} = \frac{q}{L}$$

Thermal resistivity..... $\frac{1}{k}$

Thermal resistance (degrees, per unit of heat transferred per unit time)..... R

$$R = \frac{t_1 - t_2}{q} = \frac{L}{kA}$$

Thermal conductance (heat transferred per unit time, per degree)..... C

$$C = \frac{1}{R} = \frac{kA}{L} = \frac{q}{t_1 - t_2}$$

Thermal conductance per unit area, sometimes called "Unit Conductance" (heat transferred per unit time per unit area, per degree)..... C_A

$$C_A = \frac{C}{A} = \frac{1}{RA} = \frac{q}{A(t_1 - t_2)} = \frac{k}{L}$$

Surface coefficient of heat transfer, film coefficient of heat transfer, individual coefficient of heat transfer (heat transferred per unit time, per unit area, per degree)..... h

$$h = \frac{q}{\frac{A}{t_1 - t_2}}$$

(In general h is not equal to $\frac{k}{L}$, where L is the actual thickness of the fluid film.)

Over-all coefficient of heat transfer, thermal transmittance per unit area (heat transferred per unit time per unit area, per degree over-all)..... U

$$U = \frac{q}{t_1 - t_2}$$

The thermal conductivity units in Table CXXV are written in the form:

Heat energy transfer per unit area in unit time
Temperature difference per unit length

A thermal conductivity expressed in any of the units designated in the left-hand column can be converted into any of the units designated in the headings of the columns by *multiplying* (×) by the number which is common to the row and column.

TABLE CXXV
CONVERSION FACTORS FOR VARIOUS THERMAL CONDUCTIVITY UNITS

	Cal. per sq. cm. per sec.	Kilocalorie per sq. m. per hr.	Watt per sq. cm.	B.t.u. per sq. ft. per sec.	B.t.u. per sq. ft. per hr.	B.t.u. per sq. ft. per l
	deg. Cent. per cm.	deg. Cent. per in.	deg. Cent. per cm.	deg. Fahr. per in.	deg. Fahr. per in.	deg. Fahr. per ft.
	+	+	+	+	+	+
Cal. per sq. cm. per sec. × deg. Cent. per cm.	1	360.0	4.186	0.8064	290.3	241.9
Kilocalorie per sq. m. per hr. × deg. Cent. per m.	0.002778	1	0.01163	0.002240	8.045	0.6720
Watt per sq. cm. × deg. Cent. per cm.	0.2389	86.00	1	0.1926	693.5	57.79
B.t.u. per sq. ft. per sec. × deg. Fahr. per in.	1.240	446.4	5.191	1	3600	300
B.t.u. per sq. ft. per hr. × deg. Fahr. per in.	0.0003445	0.1240	0.001442	0.0002778	1	0.08333
B.t.u. per sq. ft. per hr. × deg. Fahr. per ft.	0.004734	1.488	0.01730	0.003333	12.00	1

NOTE.—Mean calories, mean British thermal units, and absolute watts are used.

A thermal conductivity expressed in any of the units designated in the headings of the columns can be converted into any of the units designated in the left-hand column by *dividing* (÷) by the number which is common to the row and column.

Examples:

$$10 \frac{\text{B.t.u. per sq. ft. per sec.}}{\text{deg. Fahr. per in.}} = 10 \times 5.190 = 51.90 \frac{\text{watt per sq. cm.}}{\text{deg. Cent. per cm.}}$$

or

$$10 \frac{\text{B.t.u. per sq. ft. per sec.}}{\text{deg. Fahr. per in.}} = \frac{10}{0.1927} = 51.90 \frac{\text{watt per sq. cm.}}{\text{deg. Cent. per cm.}}$$

The thermal conductivity of bituminous substances may be calculated from the following formula:

$$K = \frac{0.813}{d} [1 - 0.0003(t - 32)]$$

where K = thermal conductivity in B.t.u. per hr., sq. ft., and ° F. per in.,

d = specific gravity at 60°/60° F., and

t = temperature in ° F.

The following figures have been reported for “ K ”:¹²⁴

Petroleum asphalts.....	1.2 for temperature range, 32° F. to fusing point.
Filled paving asphalts.....	4.5 to 5.2 for temperature range 50 to 90° F.
Paraffin wax.....	1.6 for temperature range 32° F. to melting-point.

Example:—The opposite faces of a slab of petroleum asphalt, 3 inches thick, are maintained at 32° and 77° F., respectively. What is the heat flow per day through each square foot of the slab?

The result is obtained as follows: $1.2 \times 24 \times 1 \times \frac{45}{3} = 432$ B.t.u.

Test 12f. A.S.T.M. Method. The following procedure has been standardized¹²⁵ for comparing the thermal conductivity of solid materials which are in the form of flat sheets; an accuracy of 10 per cent being obtained:

The thermal conductivity of a homogeneous material is the rate of heat flow under steady conditions, per unit area, and per unit temperature gradient in the direction perpendicular to the area. Thermal conductivity is expressed in watts per square centimeter per degree Centigrade per centimeter.

The principle of the method is analogous to the potentiometer method of comparing electrical resistances. A specimen of unknown conductivity is placed in series with a standard specimen of known conductivity between plates which are maintained at different temperatures. When a steady state of heat flow is attained, the respective temperature differences across the standard and the

test specimens are measured and the conductivity of the test specimen is calculated from the following relation.

$$K = K_1 \frac{L}{L_1} \times \frac{\Delta t_1}{\Delta t}$$

where K = the conductivity of the test specimen,

K_1 = the conductivity of the standard specimen,

L = the thickness of the test specimen,

L_1 = the thickness of the standard specimen,

Δt = the temperature difference of the test specimen,

Δt_1 = the temperature difference of the standard specimen.

The apparatus, consisting of a heating plate, a cooling plate, three or four thermocouples, a calibrated galvanometer or a thermocouple potentiometer, an ice bath, and at least one standard specimen, shall conform to the following requirements:

(a) Plates: The heating and the cooling plates shall be flat pieces of metal. The contact surface of each plate shall have dimensions equal to or greater than the contact surface of the specimen. The metal surface which supplies or removes the heat from the specimens shall be maintained at a temperature which is constant and uniform within 0.1°C .

NOTE.—The heating plate may be heated either electrically or by the vapor from a liquid boiling at a definite temperature. The cooling plate may be cooled by a liquid circulating through it. A platen from a steam-heated press makes a satisfactory heating or cooling plate.

(b) Thermocouples: Thermocouples shall be made from wire not larger than 0.25 mm. (0.010 in.) in diameter, or No. 30, A. w. g., one wire being of copper and the other of a suitable alloy which will give an electromotive force of about 40 microvolts per degree Centigrade. The wire shall be provided with suitable insulation of such thickness that the over-all diameter does not exceed 0.50 mm. (0.020 in.). The junctions shall be made by soldering or welding the wires. The measuring junctions of the thermocouples used for determining the temperatures at the surface of the specimens shall be soldered to, or embedded at the midpoint of sheets of pure tin, or other malleable metal, which have the same shape as the contact surface of the specimens. Each sheet of tin shall not exceed 0.50 mm. (0.020 in.) in thickness, and shall be provided with a slot in which the wires leading to the junction may be laid. No part of the thermocouple or its mounting shall exceed 0.5 mm. (0.020 in.) in thickness. The reference or cold junction

of each thermocouple shall be mounted in a glass tube not more than 2.5 mm. (0.1 in.) in diameter, which is sealed at its lower end and is immersed in a bath of crushed ice so that the junction is at least 100 mm. (4 in.) below the surface, and at least 25 mm. (1 in.) above the lower ice level.

NOTE.—The use of a separate reference or cold junction for each thermocouple is essential only when measurements are made on specimens of high graphite content or other materials which may be electrically conducting.

(c) *Galvanometer*: If a galvanometer is used on the terminals of the thermocouples it shall have a sensitivity sufficient to indicate definitely a change of temperature of 0.1°C . at the hot junction of any of the thermocouples. The deflections of the galvanometer shall be sufficiently steady and the instrument shall be calibrated to warrant an accuracy of $\pm 0.25^{\circ}\text{C}$.

(d) *Potentiometer*: If a potentiometer is used on the terminals of the thermocouples it shall be sufficiently sensitive and precise to warrant an accuracy of $\pm 0.25^{\circ}\text{C}$.

(e) *Ice Bath*: The ice bath shall consist of a Dewar flask or other well-insulated vessel filled to a depth of at least 15 cm. (6 in.) with ice and water.

Test specimens shall be in the form of flat sheets which may be square, circular, or rectangular in shape. The specimens shall not project beyond the edges of the heating or cooling plates and the total thickness of a test specimen and a standard specimen shall not exceed one-sixth of the edge of the square, the diameter of the circle or the shortest side of the rectangle. A convenient size for test specimens is approximately 20 cm. (8 in.) square and 1 cm. (0.4 in.) in thickness. Specimens should be uniform in thickness to within ± 1.5 per cent. Thickness measurements shall be made with an accuracy of 0.5 per cent at not less than ten points uniformly distributed over the surface. The average thickness shall be used for computing the thermal conductivity. When the material is soft or easily compressible, the measurement shall be made in such a way as to indicate the thickness under the pressure used in the test.

Standard specimens shall have the same shape and size as the test specimens. Any test in which the conductivity of the test specimen is found to be less than one-half or more than twice the conductivity of the standard specimen to which it was compared shall be tested again using another standard specimen having a conduc-

tivity nearer that of the specimen under test. Standard specimens may be made from materials the thermal conductivity of which is reasonably permanent and unaffected by changes in atmospheric humidity. Standard specimens shall be certified, by a standardizing laboratory, for thermal conductivity with an accuracy of ± 2 per cent.

The heating and the cooling plates and the specimens shall be placed in a horizontal position, one on top of the other. The order of assembly from the top down shall be as follows:

- (1) Heating plate;
- (2) Soft rubber sheet;
- (3) Measuring junction of a thermocouple;
- (4) Test specimen;
- (5) Measuring junction of a thermocouple;
- (6) Thin, soft rubber sheet;
- (7) Measuring junction of a thermocouple;
- (8) Standard specimen;
- (9) Measuring junction of a thermocouple;
- (10) Soft rubber sheet;
- (11) Cooling plate.

The pressure on the specimens shall be such that doubling the pressure does not change the apparent thermal conductivity by as much as 2 per cent. The assembled plates and test specimens shall be enclosed on all sides by at least 2 in. of good thermal insulating material in order to minimize the heat loss at the edges.

NOTE.—The soft rubber sheets called for in the above assembly are for the purpose of insuring good thermal contact at hard surfaces. Soft sheet rubber known as "dental dam" is a suitable and readily obtainable sheet for this purpose. One or more layers may be used at each surface to give the desired cushioning effect. When the specimens are soft and flexible these rubber sheets may be omitted, as may also one of the measuring junctions. Pressures of the order of 200 g. per sq. cm. (400 lb. per sq. ft.) are usually sufficient to insure good thermal contact of soft rubber specimens.

Temperature of the Plates: The heating and the cooling plates shall be maintained at temperatures which are constant within $\pm 0.1^{\circ}$ C. until the temperature differences across the specimens being compared are constant within $\pm 0.25^{\circ}$ C. for one hour, or $\pm 0.10^{\circ}$ C. for 30 minutes.

Temperature of Specimens: The mean of the temperatures at the two surfaces of the test specimen shall be considered the temperature of the specimen and shall be within $\pm 10^{\circ}$ C. of the tem-

perature at which it is desired to know the thermal conductivity. Similarly, the mean of the temperatures at the two surfaces of the standard specimen shall be considered its temperature.

The temperature of the standard specimen shall be within $\pm 10^{\circ}$ C. of a temperature at which its thermal conductivity is known. The temperature difference across any specimen shall not be greater than 50° C. nor less than 10° C.

The thermal conductivity of the test specimen shall be calculated by the formula given above.

NOTE 1.—The thermal conductivity of most materials increases with increasing temperature. The temperature coefficient near room temperature is usually within the range of 0.002 and 0.005 per 1° C. The change of thermal conductivity with temperature is usually near enough to a linear relation to permit the mean temperature of a specimen to be used even when large temperature differences exist.

NOTE 2.—Temperatures in the specimens being compared may be made approximately those desired by the selection of appropriate temperatures for the heating and the cooling plates, and by the insertion of sheets of thermal insulating material in the test assembly. The total thickness of these accessory sheets and of the specimens being compared should not exceed the thickness specified as the total thickness of a test specimen and a standard specimen.

NOTE 3.—A temperature of 70° C. is recommended as the temperature at which the thermal conductivity of electrical insulating materials should be measured since many such materials are designed for use at 40° C. above room temperature.

The report shall include the following:

- (a) The standard specimen used;
- (b) The thickness of the standard specimen;
- (c) The thickness of the test specimen;
- (d) The temperatures at the top and bottom of the test specimen;
- (e) The temperatures at the top and bottom of the standard specimen;
- (f) The computed conductivity of the test specimen and its mean temperature.

SPECIFIC HEAT

Test 12g. Conventional Method.¹²⁴ This may be calculated from the formula:¹²⁶

$$= \frac{1}{\sqrt{d}} (0.388 + 0.00045t)$$

in which c = specific heat in Btu. per pound per $^{\circ}$ F., or calories per gram per $^{\circ}$ C.,
 d = specific gravity at $60^{\circ}/60^{\circ}$ F., and
 t = temperature in $^{\circ}$ F.

In cases where the asphalt is mixed with various amounts of solids, such as sand, crushed rock, etc., the specific heat of the mixture may be calculated from the following expression:

$$C_m = 0.01[(100 - x)C_a + xC_s]$$

where x = per cent by weight of solids,

C = specific heat,

and the subscripts " a ," " s ," and " m " refer to asphalt, solid, and mixture, respectively. The relation: $C_s = 0.18 + 0.00006t^{\circ} \text{F.}$, may be used for the solid constituents. Values for C_a are given in Table CXXVI.

Example:—What is the specific heat at 60°F. of bituminous material containing by weight 15 per cent asphalt and 85 per cent solid materials? Table CXXVI and the above formulas give:

$$C_m = 0.01(15 \times 0.415) + (85 \times 0.184) = 0.22$$

TABLE CXXVI
SPECIFIC HEAT OF ASPHALTS

Temp. °F.	Btu./lb. °F. or cal./g °C.	Btu./gal. °F.	Temp. °F.	Btu./lb. °F. or cal./g °C.	Btu./gal. °F.
0	0.388	3.23	400	0.568	4.74
20	.397	3.31	420	.577	4.81
40	.406	3.38	440	.586	4.89
60	.415	3.46	460	.595	4.96
80	.424	3.53	480	.604	5.04
100	.433	3.61	500	.613	5.11
120	.442	3.69	520	.622	5.19
140	.451	3.76	540	.631	5.26
160	.460	3.84	560	.640	5.34
180	.469	3.91	580	.649	5.41
200	.478	3.99	600	.658	5.49
220	.487	4.06	620	.667	5.56
240	.496	4.14	640	.676	5.64
260	.505	4.21	660	.685	5.71
280	.514	4.29	680	.694	5.79
300	.523	4.36	700	.703	5.86
320	.532	4.44	720	.712	5.94
340	.541	4.51	740	.721	6.01
360	.550	4.59	760	.730	6.09
380	.559	4.66	780	.739	6.16
			800	.748	6.24

HEAT CONTENT

Test 12h. Conventional Method. This test¹²⁴ embraces the following:

The data in Table CXXVII, on the heat content of asphalts containing various percentages of mineral matter were calculated from the following equation:

$$H_a = (0.388t + 0.000225t^2 - 12.65) (1 - 0.01x) + (0.18t + 0.00003t^2 - 5.76) 0.01x$$

in which t = temperature in ° F. and

x = per cent, by weight, of mineral matter.

The data given in the column marked "0 per cent" are applicable to the bitumen content of natural asphalts and to petroleum asphalts which usually contain only small amounts of mineral matter. The data given in the columns marked "10 per cent" to "80 per cent," inclusive, are applicable to natural asphalts and to mixtures of natural or petroleum asphalts with known amounts of mineral matter. In using these data, it should be noted that the content of mineral matter, includes the so-called "free-carbon" content of the asphalt.

Example.—How much heat is required to raise the temperature of asphalt containing 10 per cent of mineral matter from 60° to 400° F.? The result may be obtained from Table CXXVII as follows:

Heat content of asphalt at 400° F. = 168 Btu./lb.

Heat content of asphalt at 60° F. = 11 Btu./lb.

Heat required = difference = 157 Btu./lb.

THERMAL EXPANSION

The thermal expansion of asphalts and other forms of bituminous matter may be calculated¹²⁴ from the values given in Table CXXVIII, where V_{60}/V represent volumes at 60° F. occupied by a unit volume at indicated temperatures, t ° F. For example, 1 gallon of petroleum asphalt measured at 350° F. will have a volume of 0.9031 gallon at 60° F.

The data given in Table CXXVIII were calculated from the equation,

$$V_t = V_{60} [1 + A(t - 60) + B(t - 60)^2]$$

TABLE CXXXVII
HEAT CONTENT OF ASPHALT IN BTU./LB.

Temp. °F.	Heat content of asphalts containing 0 to 80 per cent mineral matter, by weight								Temp. °F.
	0	10	20	30	40	50	60	80	
0	-13	-12	-11	-11	-10	-9	-8	-7	0
10	-9	-8	-8	-7	-7	-6	-6	-5	10
20	-5	-5	-4	-4	-4	-3	-3	-3	20
32	0	0	0	0	0	0	0	0	32
40	+3	+3	+3	+3	+2	+2	+2	+2	40
50	7	7	6	6	6	5	5	4	50
60	11	11	10	10	9	8	8	6	60
70	16	15	14	13	12	11	10	9	70
80	20	19	18	17	15	14	13	11	80
90	24	23	21	20	19	17	16	13	90
100	28	27	25	24	22	21	19	16	100
110	33	31	29	27	25	24	22	18	110
120	37	35	33	31	29	27	25	20	120
130	42	39	37	35	32	30	28	23	130
140	46	43	41	38	36	33	30	25	140
150	51	48	45	42	39	36	33	28	150
160	55	52	49	46	43	40	36	30	160
170	60	57	53	50	46	43	39	33	170
180	65	61	57	53	50	46	42	35	180
190	69	65	61	57	53	49	45	37	190
200	74	70	66	61	57	53	48	40	200
210	79	74	70	65	61	56	52	42	210
220	84	79	74	69	64	60	55	45	220
230	89	83	78	73	68	63	58	47	230
240	93	88	83	77	72	66	61	50	240
250	98	93	87	81	75	70	64	52	250
260	103	97	91	85	79	73	67	55	260
270	108	102	96	89	83	77	70	58	270
280	114	107	100	94	87	80	74	60	280
290	119	112	105	98	91	84	77	63	290
300	124	117	109	102	95	87	80	65	300
310	129	122	114	106	99	91	83	68	310
320	135	127	119	111	103	95	87	71	320
330	140	132	123	115	107	98	90	73	330
340	145	137	128	120	111	102	94	76	340
350	151	142	133	124	115	106	97	79	350
360	156	147	138	128	119	110	100	82	360
370	162	152	142	133	123	113	104	84	370
380	167	157	147	137	127	117	107	87	380
390	173	163	152	142	131	121	111	90	390
400	179	168	157	146	136	125	114	93	400
420	190	178	167	155	144	133	121	98	420
440	202	189	177	165	153	140	128	104	440
460	213	200	187	174	161	148	135	109	460
480	225	212	198	184	170	156	143	115	480
500	238	223	208	194	179	165	150	121	500

TABLE CXXVIII
THERMAL EXPANSION OF PETROLEUM ASPHALTS AND FLUXES

Temp. °F.	V_{60} V_t	Temp. °F.	V_{60} V_t	Temp. °F.	V_{60} V_t	Temp. °F.	V_{60} V_t	Temp. °F.	V_{60} V_t
0	1.0205	100	.9864	200	.9527	300	.9195	400	.8869
2	1.0198	102	.9857	202	.9520	302	.9188	402	.8863
4	1.0191	104	.9850	204	.9513	304	.9181	404	.8856
6	1.0185	106	.9844	206	.9506	306	.9175	406	.8850
8	1.0178	108	.9837	208	.9500	308	.9168	408	.8843
10	1.0171	110	.9830	210	.9493	310	.9162	410	.8837
12	1.0164	112	.9823	212	.9486	312	.9155	412	.8831
14	1.0157	114	.9816	214	.9480	314	.9149	414	.8824
16	1.0150	116	.9810	216	.9473	316	.9142	416	.8818
18	1.0143	118	.9803	218	.9466	318	.9135	418	.8811
20	1.0137	120	.9796	220	.9460	320	.9129	420	.8805
22	1.0130	122	.9789	222	.9453	322	.9122	422	.8799
24	1.0123	124	.9783	224	.9446	324	.9116	424	.8792
26	1.0116	126	.9776	226	.9440	326	.9109	426	.8786
28	1.0109	128	.9769	228	.9433	328	.9103	428	.8779
30	1.0102	130	.9762	230	.9426	330	.9096	430	.8773
32	1.0095	132	.9755	232	.9420	332	.9090	432	.8767
34	1.0089	134	.9749	234	.9413	334	.9083	434	.8760
36	1.0082	136	.9742	236	.9406	336	.9077	436	.8754
38	1.0075	138	.9735	238	.9400	338	.9070	438	.8747
40	1.0068	140	.9728	240	.9393	340	.9064	440	.8741
42	1.0061	142	.9722	242	.9386	342	.9057	442	.8735
44	1.0054	144	.9715	244	.9380	344	.9051	444	.8728
46	1.0048	146	.9708	246	.9373	346	.9044	446	.8722
48	1.0041	148	.9701	248	.9367	348	.9038	448	.8716
50	1.0034	150	.9695	250	.9360	350	.9031	450	.8709
52	1.0027	152	.9688	252	.9353	352	.9025	452	.8703
54	1.0020	154	.9681	254	.9347	354	.9018	454	.8697
56	1.0014	156	.9674	256	.9340	356	.9012	456	.8690
58	1.0007	158	.9668	258	.9333	358	.9005	458	.8684
60	1.0000	160	.9661	260	.9327	360	.8999	460	.8678
62	.9993	162	.9654	262	.9320	362	.8992	462	.8671
64	.9986	164	.9647	264	.9313	364	.8986	464	.8665
66	.9980	166	.9641	266	.9307	366	.8979	466	.8659
68	.9973	168	.9634	268	.9300	368	.8973	468	.8652
70	.9966	170	.9627	270	.9294	370	.8966	470	.8646
72	.9959	172	.9620	272	.9287	372	.8960	472	.8640
74	.9952	174	.9614	274	.9280	374	.8953	474	.8633
76	.9945	176	.9607	276	.9274	376	.8947	476	.8627
78	.9939	178	.9600	278	.9267	378	.8940	478	.8621
80	.9932	180	.9594	280	.9260	380	.8934	480	.8614
82	.9925	182	.9587	282	.9254	382	.8927	482	.8608
84	.9918	184	.9580	284	.9247	384	.8921	484	.8602
86	.9912	186	.9573	286	.9241	386	.8914	486	.8595
88	.9905	188	.9567	288	.9234	388	.8908	488	.8589
90	.9898	190	.9560	290	.9228	390	.8901	490	.8583
92	.9891	192	.9553	292	.9221	392	.8895	492	.8577
94	.9884	194	.9547	294	.9214	394	.8888	494	.8570
96	.9878	196	.9540	296	.9208	396	.8882	496	.8564
98	.9871	198	.9533	298	.9201	398	.8876	498	.8558
100	.9864	200	.9527	300	.9195	400	.8869	500	.8552

using $A = 0.000341$ and $B = 0.0000001$, which is equivalent to the following:

Temperature Range in ° F.	Mean Coefficient of Expansion
60 to 150.....	0.00035
60 to 250.....	0.00036
60 to 350.....	0.00037
60 to 450.....	0.00038

These coefficients and the expansions, $(V_{60}/V_t) - 1$, obtained from Table CXXVIII apply to petroleum asphalts and fluxes in general with an estimated accuracy of 5 per cent, which is equivalent to the following percentage accuracy in the relative volumes, V_{60}/V_t , for various temperature ranges: 0.1 per cent, 0° to 100°; 0.2 per cent, 100° to 200°; 0.4 per cent, 200° to 300°; 0.6 per cent, 300° to 400°; and 0.8 per cent, 400° to 500° F.

Products containing wax, gas bubbles, or nonbituminous materials have expansions which differ from those given, in proportion to the amounts present.

Example.—If the volume of a given quantity of petroleum asphalt is 10,000 gallons at 350° F., what is its volume at 60° F.? Calculate from the value given in Table CXXVIII as follows:

$$V_{60} = 10,000 \times 0.9031 = 9031 \text{ gallons at } 60^\circ \text{ F.}$$

The following coefficients of expansion per ° C. have been reported for various asphaltic products:¹²⁷

	Temperature Range	Coefficient of Expansion
Asphalt saturant for prepared roofing (fusing-point 34° C., K. and S.).....	15-60° C.	0.00061
Asphalt coating for prepared roofing (fusing-point 73° C., K. and S.).....	15-60° C.	0.00059
Residual petroleum asphalt (fusing-point 81° C.)	15-60° C.	0.00062
Refined Trinidad asphalt.....	15-60° C.	0.00046

Additional figures applicable to asphalts, tars and pitches are given in Table CXXIX.¹²⁸

Table CXXX has been devised for reducing the volumes of coal tar and coal-tar pitch to the basis of 60° F., and shows the volume occupied at 60° F. by a quantity of material occupying unit volume

TABLE CXXIX

COEFFICIENTS OF EXPANSION OF TYPICAL BITUMINOUS SUBSTANCES

Material		Temperature Range, deg. Fahr.	Coefficient of Expansion per 1 F.	
Crude tars....	Horizontal coal tar.....	60 to 150	0.00027	to 0.000305
	Vertical coal tar.....	60 to 150	0.000355	to 0.00037
	Coke oven tar.....	60 to 150	0.00032	to 0.00034
	Low temperature tar.....	60 to 150	0.00042	
	Light water-gas tar.....	60 to 150	0.00035	to 0.00037
	Heavy water-gas tar.....	60 to 150	0.00031	to 0.00032
Tar road materials and pitch....	<div>Coal Tar</div> <div>Water-gas Tar</div>			
	Cutback products.....	60 to 180	0.00033 to 0.00037	0.00034 to 0.00035
	Patching materials.....	60 to 180	0.00033	0.00328 to 0.00034
	Hot application.....	100 to 200	0.0003 to 0.00035	0.00029 to 0.00032
	Cements.....	100 to 300	0.0003	0.0003
Asphaltic road material....	Pitches.....	200 to 300	0.000255 to 0.00028	0.000255 to 0.00028
	Road oils.....	60 to 180	0.0004 to 0.00041	
	Cutbacks.....	60 to 180	0.0004 to 0.00042	
	Hot application.....	200 to 300	0.00036	
Solid asphalts.....	Cements.....	200 to 350	0.00033 to 0.00034	
		42 to 140	0.00035 to 0.00039	
		60 to 250	0.00033 to 0.00039	
		200 to 400	0.00033 to 0.00035	
		250 to 350	0.000362 to 0.000384	
Petroleum asphalts and fluxes		350 to 450	0.000378 to 0.000399	
		60 to 450	0.00036 to 0.000382	
		60 to 150	0.00035	
		60 to 250	0.00036	
		60 to 350	0.00037	
		60 to 450	0.00038	

at the indicated temperature.¹²⁰ The groups and coefficients of expansion for the several subdivisions of the table are as follows:

Group Number	Coefficient of Expansion, per Deg. Fahr. at 60° F.	Material
000	0.00026.....	{ Coal-Tar Pitch for Roofing, Dampproofing, and Waterproofing Coal-Tar Pitch for Stone Block Filler
00	0.00030.....	
0	0.00035.....	{ Tar, Grades T-5, T-6, T-7, T-8, T-9, T-10, T-11, T-12, T.C.B.-5, and T.C.B.-6 Tar, Grades T-1, T-2, T-3, and T-4

Similar correction tables have been published for coal-tar and creosote mixtures,¹⁸⁰ also petroleum oils.¹⁸¹

Test 12i. A.S.T.M. Method. The method on page 1041 has been standardized¹⁸² for ascertaining the coefficient of expansion of bituminous compounds.

TABLE CXXX (Continued)

Group o

Legend: t = observed temperature in degrees Fahrenheit; M = multiplier for reducing volumes to the basis of 60° F.

t	M	t	M	t	M	t	M	t	M	t	M	t	M	t	M	t	M	t	M
0	1.0211	25	1.0123	50	1.0035	75	0.9948	100	0.9862	125	0.9775	150	0.9691	175	0.9606	200	0.9533	225	0.9441
1	1.0208	26	1.0119	51	1.0032	76	0.9944	101	0.9858	126	0.9772	151	0.9687	176	0.9603	201	0.9530	226	0.9438
2	1.0204	27	1.0116	52	1.0028	77	0.9941	102	0.9855	127	0.9768	152	0.9684	177	0.9600	202	0.9517	227	0.9435
3	1.0201	28	1.0112	53	1.0025	78	0.9938	103	0.9852	128	0.9765	153	0.9680	178	0.9596	203	0.9513	228	0.9432
4	1.0197	29	1.0109	54	1.0021	79	0.9934	104	0.9848	129	0.9762	154	0.9677	179	0.9593	204	0.9510	229	0.9428
5	1.0194	30	1.0106	55	1.0017	80	0.9931	105	0.9844	130	0.9758	155	0.9674	180	0.9590	205	0.9507	230	0.9425
6	1.0190	31	1.0102	56	1.0014	81	0.9927	106	0.9841	131	0.9755	156	0.9670	181	0.9586	206	0.9504	231	0.9422
7	1.0186	32	1.0098	57	1.0010	82	0.9924	107	0.9837	132	0.9751	157	0.9667	182	0.9583	207	0.9500	232	0.9419
8	1.0183	33	1.0095	58	1.0007	83	0.9920	108	0.9834	133	0.9748	158	0.9664	183	0.9580	208	0.9497	233	0.9415
9	1.0179	34	1.0092	59	1.0003	84	0.9917	109	0.9831	134	0.9745	159	0.9660	184	0.9576	209	0.9494	234	0.9412
10	1.0176	35	1.0088	60	1.0000	85	0.9914	110	0.9827	135	0.9741	160	0.9657	185	0.9573	210	0.9490	235	0.9409
11	1.0172	36	1.0084	61	0.9997	86	0.9910	111	0.9823	136	0.9738	161	0.9654	186	0.9569	211	0.9487	236	0.9406
12	1.0168	37	1.0081	62	0.9993	87	0.9907	112	0.9820	137	0.9735	162	0.9650	187	0.9566	212	0.9484	237	0.9402
13	1.0165	38	1.0077	63	0.9990	88	0.9903	113	0.9816	138	0.9731	163	0.9647	188	0.9563	213	0.9481	238	0.9399
14	1.0161	39	1.0074	64	0.9986	89	0.9900	114	0.9813	139	0.9728	164	0.9643	189	0.9559	214	0.9477	239	0.9396
15	1.0158	40	1.0070	65	0.9982	90	0.9896	115	0.9809	140	0.9724	165	0.9640	190	0.9556	215	0.9474	240	0.9392
16	1.0154	41	1.0067	66	0.9979	91	0.9892	116	0.9806	141	0.9721	166	0.9637	191	0.9553	216	0.9471	241	0.9389
17	1.0151	42	1.0063	67	0.9976	92	0.9889	117	0.9802	142	0.9718	167	0.9633	192	0.9549	217	0.9468	242	0.9386
18	1.0147	43	1.0059	68	0.9972	93	0.9886	118	0.9799	143	0.9714	168	0.9630	193	0.9546	218	0.9465	243	0.9383
19	1.0144	44	1.0056	69	0.9969	94	0.9882	119	0.9795	144	0.9711	169	0.9627	194	0.9543	219	0.9461	244	0.9380
20	1.0141	45	1.0052	70	0.9965	95	0.9879	120	0.9792	145	0.9707	170	0.9623	195	0.9539	220	0.9458	245	0.9376
21	1.0137	46	1.0049	71	0.9962	96	0.9876	121	0.9789	146	0.9704	171	0.9620	196	0.9536	221	0.9455	246	0.9373
22	1.0134	47	1.0045	72	0.9958	97	0.9872	122	0.9785	147	0.9701	172	0.9616	197	0.9533	222	0.9452	247	0.9370
23	1.0130	48	1.0042	73	0.9955	98	0.9869	123	0.9782	148	0.9697	173	0.9613	198	0.9530	223	0.9449	248	0.9367
24	1.0126	49	1.0039	74	0.9952	99	0.9865	124	0.9779	149	0.9694	174	0.9610	199	0.9527	224	0.9445	249	0.9364

NOTE.—Certain materials when cooled form voids within the mass and no satisfactory method has been found to measure their coefficient of expansion.

This method is intended for determining the true and effective coefficient of expansion of compounds. The true coefficient of expansion is for compounds which are free of entrapped gases. The

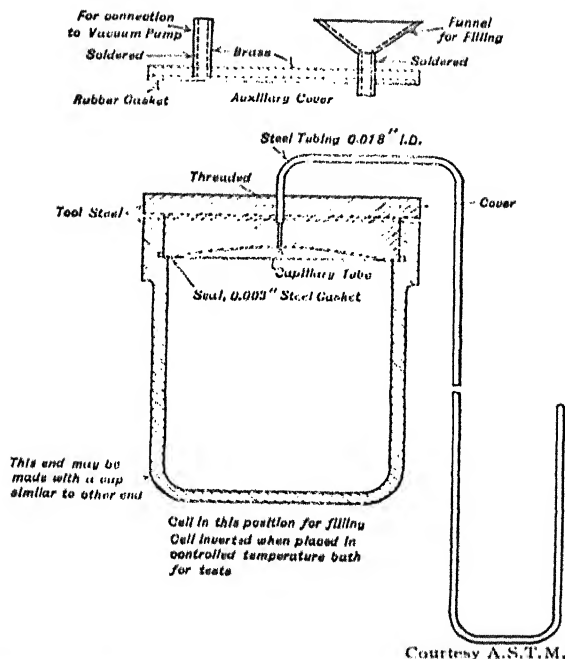


FIG. 264.—Metallic Cell for Coefficient of Expansion Determinations.

effective coefficient of expansion is the coefficient for material which has not been degasified just prior to test. It is important for many purposes to know the effective coefficient of the material as received or after heating to the maximum temperature of application. Consistent results, however, may only be obtained with gas-free compounds.

Compounds having high viscosity may be degasified by heating in a vacuum oven or in a cell fitted with a special cover with vacuum connection similar to the cover shown in Fig. 264.

In either method the temperature and vacuum should be high enough and the time long enough to insure the driving off of mechanically entrapped gases, but should tend to decompose the compound as little as possible. A gas-free condition is indicated when bubbles of gas no longer appear at the surface of the compound.

The percentage of entrapped gases may be calculated from the following formula (See also Test 7f):

Volume, in per cent, of entrapped gases

$$= \frac{\text{sp. gr. of degasified compound} - \text{sp. gr. of untreated compound}}{\text{sp. gr. of degasified compound}} \times 100$$

The following methods are recommended:

Methods A and B. Methods *A* and *B* for true coefficient of expansion are intended for use only where the uniformity of the material under test justifies a high degree of precision. Method *A* is suitable for testing low-viscosity types such as waxes and petrolatums. Method *B* is suitable for testing asphalts and high-viscosity materials, also for opaque materials which may give difficulty in reading the glass scale of Method *A*.

Methods C, D and E. Methods *C, D* and *E* are intended for faster testing where high precision may not be justified. These methods may be used for determining either true or effective coefficient of expansion.

Method A. Using Glass Flask

(a) *Flask:* The flask* shall hold approximately 250 ml. to the zero mark, and the neck of the flask shall be 1 cm. in internal diameter and graduated for 25 ml. in 0.1-ml. divisions.

(b) *Oil Bath:* The cylindrical oil bath for heating the sample shall be approximately 10 in. (25.4 cm.) in inside diameter and 20 in. (50.8 cm.) in inside depth with a false bottom 1 in. from the bottom and shall have provision for circulating and heating the oil.

(c) *Metal Collar:* Lead or iron collars shall be used on the neck of the flask during test to prevent the oil currents of the bath from moving the flask.

* A Pyrex or quartz glass flask is very satisfactory for this purpose.

The capacity of the flask at the zero point and several points on the scale shall be determined by filling the flask with distilled water at a known temperature and weighing.

The flask shall be maintained under a vacuum of 25 in. of mercury and a temperature 50° C. higher than the softening point (Ring-and-ball method) while being filled and for approximately 30 min. after filling is complete. The flask shall be filled to within the last milliliter marked on the neck when held at the maximum test temperature and slowly cooled to room temperature (10 to 12 hr.). Before starting the test, the flask shall be examined for the presence of cavities or irregular contraction of the compound. Some compounds after cooling below the liquid state, tend to stick to the sides of the neck of the flask. In such cases it is necessary to gradually warm the neck and flow the compound to meet the rest, after which the flask shall be placed in the bath for several hours to insure temperature equilibrium.

With the compound satisfactorily placed in the flask at the lowest temperature, the height of the column in the neck shall be read and the bath then slowly heated. Readings shall be taken at 50° C. intervals, holding the bath as constant as possible at each point until no more expansion occurs at that point, the procedure being repeated for each point until maximum temperature is reached.

During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of the expansion of the compound shall be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction it may be reduced to 30 min.

After securing the readings over the temperature range desired, a curve shall be plotted from the temperature and expansion readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{V_1 - V}{(T_1 - T)V} + C$$

where V = the original volume occupied by the compound,

V_1 = the volume at higher temperature occupied by the compound,

T = the original temperature,

T_1 = the higher temperature, and

C = a constant = coefficient of expansion of glass container.

* The coefficient of expansion shall be calculated for three temperature ranges, as follows:

(1) From the minimum temperature at which the measurement was made to 10°C. below the melting point. This is intended to give the average coefficient for the solid condition.

(2) From 5°C. above the melting point to 100°C. This is intended to give the average coefficient for the liquid condition.

(3) From the minimum temperature at which a measurement was made to 100°C.

Method B. Using Metallic Cell

(a) Metal Cell: The cell shall be made of steel, consisting of four parts, a cylinder about 2.5 in. in internal diameter, having a rigid bottom, a metallic gasket, a cover to which a steel capillary tube is attached. The cell shall have an internal volume of approximately 250 ml.

Figure 264 shows the metallic cell for coefficient of expansion determinations. The cell consists of four principal parts: a steel cylinder, a metallic gasket, a steel cover, and a dummy or auxiliary cover for filling. The gasket must be of a metal which does not amalgamate with mercury.

The cylinder is about 2.5 in. (6.35 cm.) in internal diameter, and approximately 3 in. (7.6 cm.) in internal depth. The top of the cylinder is threaded to receive the steel cover and has a machined shoulder to seat a 0.003 in. thick metallic gasket. The cylinder may be of one piece construction or fitted with a cap at the bottom similar to the top end. The steel cover is carefully rounded on the under side to avoid air pockets. It is threaded into the top of the cylinder and seats on the metallic gasket. The center of the cover is threaded to receive a steel capillary tube of 0.018 in. in internal diameter. The auxiliary cover is brass with a rubber gasket seated by atmospheric pressure. A brass funnel and vacuum connection are soldered to the cover. A plug is provided for seal-

ing the funnel while the cell is maintained under the vacuum specified after filling.

(*b*) Oil Bath: The oil bath shall be the same as described in Method *A*, with the exception that provision shall be made for supporting the metal cell.

The cell shall be calibrated to determine its volume at various temperatures as follows:

- (1) Weigh the assembled cell to determine its tare weight.
- (2) Fill the cell with mercury until replacing the cover causes some to extrude through the capillary tubing. Record the weight of the cell and mercury and note the temperature.
- (3) Place cell in the oil bath in an inverted position. The capillary tubing should extend over the side of the oil bath in such a way that the extruded mercury may be caught in a beaker. The oil bath, which is several degrees above room temperature causes some mercury to be extruded from the capillary tube. When all expansion has taken place, weigh the mercury collected.
- (4) Adjust the oil bath for other test temperatures and note the amounts of mercury extruded. The weight of mercury in the cell at any temperature is thus determined and the volume may be calculated.

While filling the cell it shall be placed in an oil bath and maintained at a temperature 50° C. higher than the softening point of the compound (Ball-and-shouldered-ring method, as determined). When the cell has been filled to within $\frac{1}{4}$ in. of the cover it shall be placed in a vacuum oven and maintained at a vacuum of 25 in. of mercury and a temperature 50° C. higher than the softening point of the compound for a period of not less than 30 min. nor more than 45 min. At the end of this period the cell shall be slowly cooled to room temperature, and any irregularities in the surface of the compound removed. The cover shall then be screwed on and the cell and compound weighed again.

Sufficient mercury shall be poured into the cell so that some is extruded when the cover is screwed down. The cell shall then be weighed again. The cell shall then be inverted and placed in the oil bath, and the procedure outlined under calibration, Method *B* again followed for 5° C. intervals. Only clean, distilled mercury shall be used. During the test, temperature readings shall be taken at top and bottom of the bath to detect any variation. Readings of

the expansion of the compound should be made at intervals long enough to insure uniform temperature distribution and complete movement of the compound. Until complete liquefaction, the interval should be 3 to 4 hr.; after liquefaction, it may be reduced to 30 min.

After volumetric determinations have been made over the desired temperature range, a curve shall be plotted between volume and temperature readings from which the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{V_1 - V}{(T_1 - T)V}$$

where V = the original volume occupied by the compound,
 V_1 = the volume at higher temperature occupied by the compound,
 T = the original temperature, and
 T_1 = the final temperature.

The coefficient of expansion shall be calculated for the same three ranges as prescribed in Method A.

Method C. Specific Gravity Method

The specific gravity of untreated or degasified compound at two test temperatures shall be ascertained by any of the suitable procedures described in Test 7. From the temperature and specific gravity the coefficient of expansion shall be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{\text{sp. gr. at } T - \text{sp. gr. at } T_1}{(T_1 - T) \text{ sp. gr. at } T_1}$$

where T = the initial temperature, and
 T_1 = the higher temperature.

Method D. Pyknometer Contraction

This method is a modification of the specific gravity method (Method C). It may be applied to either untreated or degasified compounds.

(a) *Flask*: A 100-ml. volumetric glass * flask shall be used and the zero mark shall be as near as possible to the bulb of the flask.

(b) *Oil Bath*: The oil bath may consist of a tall-form glass * beaker of sufficient size so that when the flask is supported about 1 in. from the bottom the oil level will reach at least to the zero mark of the flask.

(c) *Metal Collar*: Lead or iron collars placed on the neck of the flask shall be used during heating to prevent oil currents of the bath from moving the flask.

The flask shall be weighed and then filled to within approximately 10 ml. of the zero mark with the compound to be tested, care being taken that at no time shall the temperature of the compound exceed the softening point (shouldered ring-and-ball method) by more than 10° C. Care shall also be taken so that none of the compound remains in the stem of the flask. The flask shall be cooled to room temperature, weighed, immersed in the oil bath and placed in an oven previously heated to the maximum temperature at which the test is to be conducted. After the oil bath reaches the test temperature it shall be allowed to remain at this temperature for 1 hr. The flask shall be removed from the oven, and mercury from a burette shall be quickly poured into it until the level of the compound is within 0.5 ml. of the zero mark. The quantity of mercury used shall be noted in milliliters. The flask shall be replaced in the oven for 20 min., removed again and the mercury titration shall be continued until the level of the compound is exactly at the zero mark. The number of milliliters of mercury used in both titrations shall be recorded.

The oven temperature shall be reset at the next lower test point, temperature equilibrium allowed for as above, and the titration shall be repeated. The same procedure shall be continued at all subsequent test points, the milliliters of mercury added at each point being recorded.

To find the coefficient of expansion of the material between any two temperatures, the calculations shall be made as follows:

* Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

Volume of mercury A at room temperature corrected to temperature $t = A + 0.000182A(t - 20)$.

Example.—If volume of flask is 100 ml. at 20°C ., volume at $t = 100 + 0.0025(t - 20)$ ml.

Let $X = V_1 - M_1 =$ the volume of compound at T_1 ,

Let $Y = V_2 - M_2 =$ the volume of compound at T_2 ,

Then the expansion in cu. cm. per gram, $T_1 - T_2 = \frac{X}{W} - \frac{Y}{W'}$

the expansion per cu. cm., $T_1 - T_2 = \frac{\frac{X - Y}{W'}}{\frac{Y}{W}} = \frac{X - Y}{Y}$

the coefficient of expansion, cu. cm. per cu. cm. $= \frac{X - Y}{Y(T_1 - T_2)}$ per degree Centigrade.

where $W =$ the weight of sample in grams,

$T_1 =$ the higher temperature in degrees Centigrade,

$T_2 =$ the lower temperature in degrees Centigrade,

$M_1 =$ milliliters of mercury (corrected) added at T_1 ,

$M_2 =$ the sum of milliliters of mercury (corrected) added at T_1 and at T_2 ,

$V_1 =$ the corrected volume of flask at T_1 ,

$V_2 =$ the corrected volume of flask at T_2 .

To obtain the curve of expansion, plot the points $\frac{W}{X}$, $\frac{W}{Y}$, etc., which are the densities at various temperatures.

Method E. Pyknometer Expansion

This method is another modification of the specific gravity method (Method C) and may also be applied to either untreated or degasified materials.

The pyknometer and bath required are the same as described in Method D, except that the neck of the flask shall be cut off at the 100-ml. point and ground square. A metal pyknometer may be used provided its coefficient of expansion is known. In this procedure the pyknometer shall be allowed to cool slowly to the lowest test temperature. During the cooling period the flask shall be kept filled by adding more compound and after equilibrium is reached the excess material shall be removed by passing a sharp, flat blade over the rim. The flask shall then be removed from the bath and quickly weighed. Knowing the tare weight and volume of the flask, the specific gravity may be determined. For successively higher temperatures, it is only necessary to weigh the extruded portion.

From the temperature and weight readings the coefficient of expansion may be calculated from the following formula:

$$\text{Coefficient of expansion} = \frac{W - W_1}{W_1(T_1 - T)} - \frac{WC}{W_1}$$

where W = the initial weight of the compound in the flask,

W_1 = the weight of the compound in the flask at higher temperature,

T = the initial temperature,

T_1 = the higher temperature,

C = the coefficient of expansion of the flask.

Method F. Plummets Displacement

This method is another modification of the specific gravity method (Method C).

The apparatus shall consist of the following:

- (a) Balance: An analytical balance equipped with pan straddle.
- (b) Plummets: An aluminum plummet of suitable shape weighing 5 to 10 g.
- (c) Beaker: A 400-ml. glass * beaker wrapped with asbestos.
- (d) Thermometer: A thermometer of suitable range.
- (e) Wire: Two pieces of fine copper wire.

The specific gravity at pouring temperature shall be calculated from the weight of compound displaced by the calibrated aluminum plummet.

Calibration of Plummet. The following weight determinations of the plummet, to the nearest 1 mg., shall be made:

a = the weight in air;

b = the weight suspended in water at 25° C. (77° F.).

Then: $a - b$ = the weight of water displacement at 25° C. (77° F.).

The value of the plummet displacement (D_{tp}) in terms of grams of water at 25° C. (77° F.) shall then be corrected to the

* Pyrex glass or other material with equally low coefficient of expansion is very satisfactory for this purpose.

pouring temperature, t_p , in Centigrade degrees by means of the following formula:

$$D_{tp} = 0.000076(t_p - 25)(a - b) + (a - b)$$

NOTE.—Coefficient of cubical expansion of aluminum per deg. Cent. = 0.000076.

Testing of the Sample. The sample shall be carefully melted in the beaker and the temperature raised to approximately 15° C. (27° F.) above the desired test temperature. The beaker shall be placed on the straddle and the plummet suspended in the compound by the fine copper wire. (The weight of the wire should be balanced by placing an equal weight of the wire on the opposite balance pan.)

The scales shall be balanced approximately and at the same time the sample shall be stirred slowly, using the thermometer as a stirring rod. When the sample has cooled to the desired temperature, the weighing shall be completed rapidly.

Calculation of Specific Gravity, $t_p/25^\circ$ C. The specific gravity shall be calculated from the following formula:

$$\text{Sp. Gr., } t_p/25^\circ \text{ C.} = \frac{\text{Weight of Plummet in Air} - \text{Weight of Plummet in Compound}}{D_{tp}}$$

After the specific gravity of the sample at 25° C. (77° F.), and at pouring temperatures have been determined according to the above described methods, the coefficient of expansion shall be calculated according to the formula given in Method C.

BREAKING POINT

Various devices have been recommended for ascertaining the breaking-point of asphalts and pitches.¹³³ They all are based upon determining the temperature at which a layer of predetermined thickness assumes a definite degree of brittleness. The test is of value in determining the temperature range at which bituminous materials in a comparative thin layer will remain pliable to the extent of bending without breaking or fracturing.

Test 13a. Knife Test. The following simple test has been standardized for ascertaining the breaking-point of bituminous compositions:¹³² The substance shall be spread on a piece of sheet metal in a layer $\frac{1}{16}$ in. in thickness. It shall then be submerged in

water having a temperature at least $20^{\circ}\text{F}.$ above the expected breaking-point. It shall then be cooled $5^{\circ}\text{F}.$ and held at this temperature for 5 minutes. The cooling shall then be continued in increments of $5^{\circ}\text{F}.$ At each temperature interval the substance shall be tested with a blade of a knife, inserting the point with the blade as nearly parallel with the sheet metal as is practicable, and pulling the substance from the sheet metal as rapidly as possible by rotating the blade about its back as an axis. The temperature at which the substance no longer stretches but snaps, shall be considered the breaking-point.

Test 13b. Reeve and Yeager's Method. The following procedure has been proposed by Reeve and Yeager:¹⁸⁴ A standard test piece of bituminous material $\frac{1}{4}$ in. thick, 1 in. wide, and 4 in. long, is prepared by melting at as low a temperature as possible to avoid volatilization and then pouring into a brass mold coated with mercury. The specimen is carefully removed and inserted into the clamp of the apparatus illustrated in Fig. 368. The water bath is maintained at a temperature somewhat above the estimated breaking-point for fifteen minutes, whereupon the sample is bent around the mandrel of $\frac{3}{16}$ -in. radius, through an arc of 180 deg. in exactly ten seconds, using a metronome. If not broken, another specimen is inserted in the clamp and the temperature of the bath lowered $10^{\circ}\text{F}.$ and the test repeated. In this way, working between limits and gradually narrowing them, a point is reached at which a change of $2^{\circ}\text{F}.$ shows a break or no break. The lower temperature is then reported as the "breaking-point" of the material under examination.

Test 13c. Fraas Method. The following test has been proposed by Ing. A. Fraas,¹⁸⁵ in which the breaking-point represents the temperature at which a layer of the substance 0.5 mm. thick affixed to a sheet of steel, will crack when bent over a mandrel 9 mm. in diameter in one second time. The prescribed quantity of substance for each test is 0.4 ml., which may either be weighed directly on the steel strip, or extruded from the small press "A" shown in Fig. 265, the details of which are illustrated in Fig. 266, of which the mold is cylindrical in form, measuring 20 mm. in diameter by 20 mm. in height, having a slit in the bottom 20 mm. long by 0.5 mm. wide, through which is extruded a 0.4 ml. strip of stand-

ard dimensions. The strip is applied to a sheet of terne steel 40 mm. by 20 mm. by 0.15 mm. and heated at 105–110° C. until air

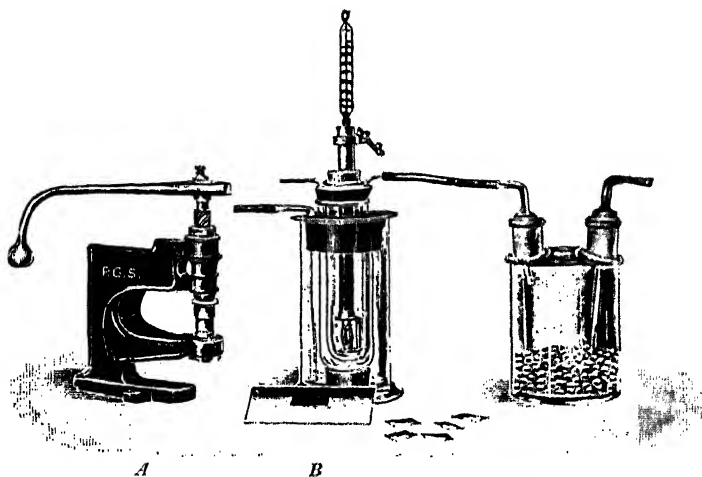


FIG. 265.—Fraas Breaking-point Apparatus.

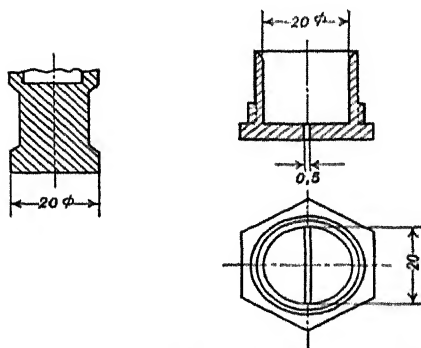


FIG. 266.—Mold of Fraas Breaking-point Apparatus.

and moisture are expelled and proper adhesion to the metal is secured. Any minute air bubbles are removed from the surface by the rapid passage of a gas flame over same. When cool, the strip

is inserted in the clip at the lower end of the apparatus "B" shown in Fig. 265, which in turn is introduced in a large test-tube supported in a transparent vacuum-flask containing the cooling me-

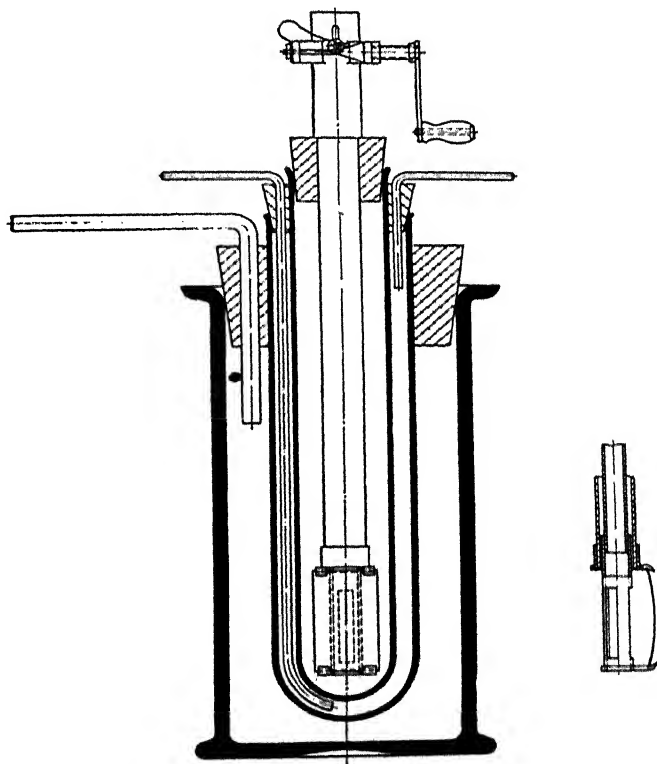


FIG. 267.—Fraas Breaking-point Tester Shown Sectionally.

dium. The apparatus as assembled is illustrated sectionally in Fig. 267. Cooling may be effected by drawing dry air through ether, or for lower temperatures (i.e., to minus 20° C.) through a mixture of solid CO_2 with alcohol or ether. The rate of temperature fall is carefully adjusted to 1° C. per minute, and the specimen is bent at each degree drop in temperature (commencing at 10° C. above the expected breaking-point) by turning the handle at the top

of the apparatus, at a speed of one revolution per second. This causes the specimen to be bent around the mandrel 9 mm. in diameter, whereupon the handle is returned to its original position. The temperature at which the bituminous substance is first observed to crack through to the metal is recorded as the "breaking-point." Duplicate tests are performed, and the lower reading is taken. The test is claimed to be accurate to $\pm 1^{\circ}$ C.

SOLIDIFYING-POINT

Test 14. Metzger's Method. The solidifying-point as proposed by Hans Metzger¹⁰⁰ represents the temperature at which the

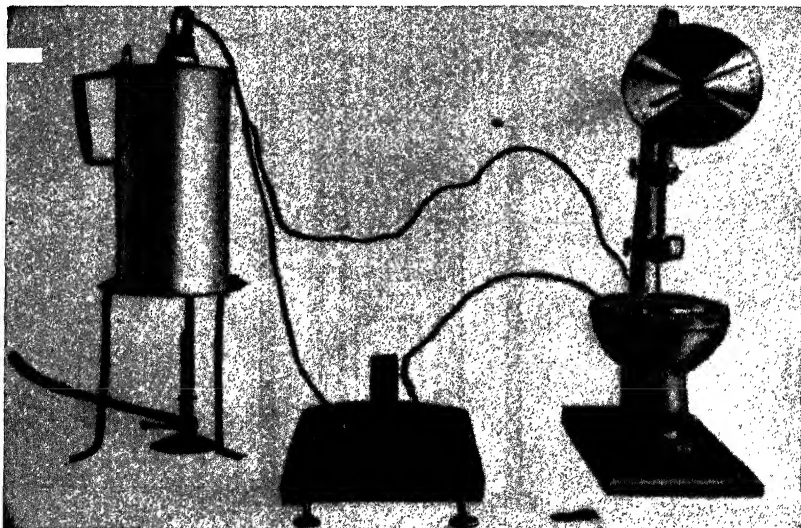


FIG. 268.—Metzger Solidifying-point Tester Assembled.

substance upon cooling attains a definite degree of solidity (i.e., hardness). Specifically, it represents the temperature at which a cylindrical plunger 0.5 mm. in diameter will penetrate the bituminous substance exactly 0.1 mm. under a load of 450 g. in 60 seconds. The apparatus for ascertaining the solidifying-point is illustrated in Figs. 268, 269 and 270, and is composed of a plunger "q" having a flat bottom 0.5 mm. in diameter, attached to the lower

end of a shaft "n," which with the weight "o" aggregates 450 g. The motion of the shaft is measured by the milled rod "c," connected by suitable gears in the housing "b" with the pointer "k" on the movable scale "i," graduated in degrees and tenths of a degree, corresponding to penetrations of 0.1 mm. and 0.01 mm. re-

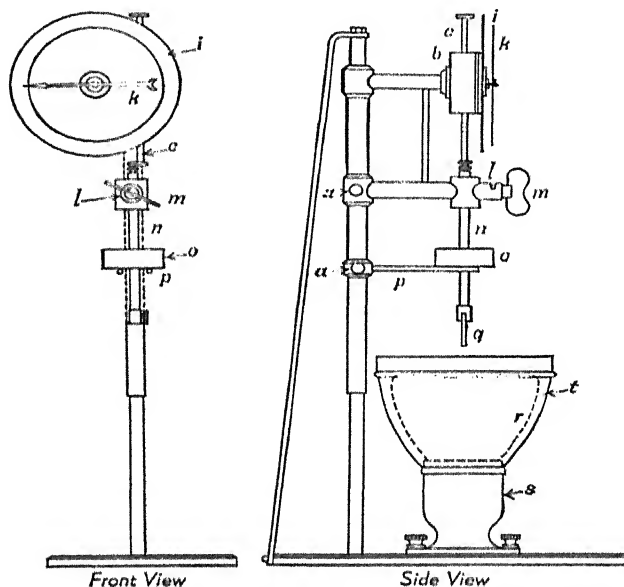


FIG. 269.—Metzger Solidifying-point Tester Shown Sectionally.

spectively. The shaft is held in position by the winged screw "m" actuating against the clamp "l." The fork "p" is used to support the weight and attached shaft when the instrument is not in use.

The substance to be tested is melted at the lowest possible temperature and poured into the receptacle "u," measuring 1.5 cm. wide, 3 cm. long and 2 mm. deep. Before it solidifies, the junction of a thermo-couple composed of iron and Constantine wires is embedded below the surface of the substance, in which it is caused to protrude 1 cm. The receptacle is supported horizontally upon being slid on the runners of the support "v" cemented inside a Dewar vessel "r," enclosed in a metal jacket "t," which in turn is attached

to the base "s." One end of the thermo-couple, together with a thermometer, is immersed in paraffin oil maintained at a constant temperature of 35°C . in a stoppered Thermos flask, which in turn is enclosed in a metal vessel heated with a gas flame, regulated by a thermostat, the purpose of which is to prevent radiation from the

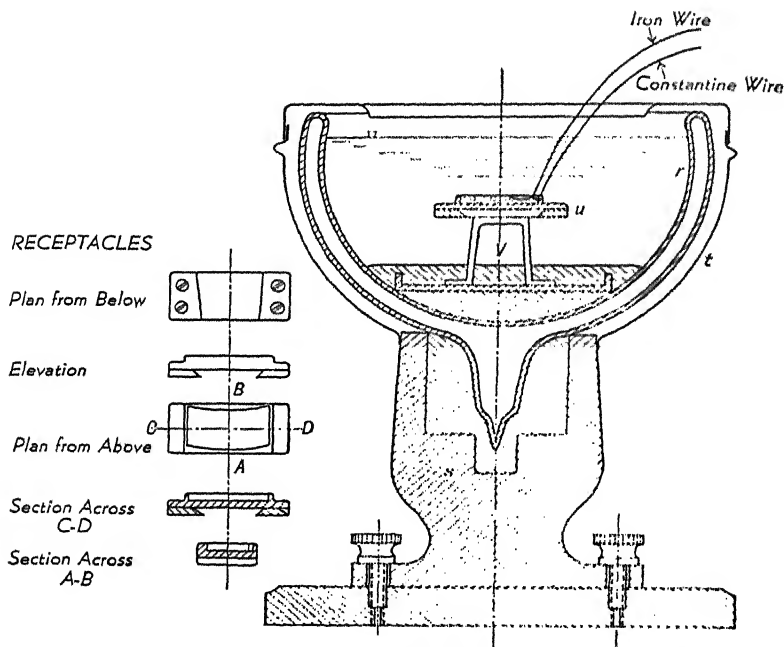


FIG. 270.—Details of Receptacle—Metzger's Solidifying-point Tester.

Thermos flask. The free ends of the wires are connected with a galvanometer (198 ohms) reading to 5 millivolts, which under the prescribed conditions will record temperatures as low as -70°C . The galvanometer readings should be accurately calibrated against a pentane thermometer in tenths of a degree.

The Dewar vessel "r" is thereupon filled with refrigerated alcohol to within 1 cm. of its upper rim, which is further cooled by stirring in solidified carbon-dioxide in powdered form until the temperature of the substance is brought about 10°C . lower than the

estimated solidifying-point. When the galvanometer reading remains substantially constant, the measuring device is lowered by releasing the screws "a" until the plunger "q" is supported within 2 mm. of the surface of the substance. Then by releasing the winged screw "m," the plunger is carefully lowered until it rests against its surface. The rod "c" is thereupon depressed until its lower foot is brought in contact with the shaft, when the scale "i" is rotated until set at the zero point. Using a stop-watch, the shaft is suddenly released and the plunger allowed to penetrate the substance for exactly 60 seconds, during which interval it is maintained within a temperature range of 1 to 2° C., whereupon a reading is taken to ascertain the depth of penetration.

The temperature of the alcohol is raised slightly, by stirring or blowing air through it, and when it becomes constant, another penetration reading is taken 2 to 3 mm. from the first spot (upon rotating the Dewar vessel). In this manner, a series of temperature and penetration readings are obtained, until the penetration finally measures 0.3 to 0.4 mm. From these readings a graph is constructed on coordinate paper, in which the penetration depths are recorded as abscissas and the temperature as ordinates. By interpolation, the temperature is noted at which the penetration equals exactly 0.1 mm., and this temperature is recorded as the "solidifying-point."

Metzger has worked out some interesting relationships between the solidifying-point representing the temperature at which the bituminous substance attains a *definite* degree of solidity, and Ubbelohde's liquefying-point (Test 15*h*)—representing the temperature at which the substance attains a definite degree of liquidity (i.e., softness).

These tests are taken as the two extremes, representing fixed points in the consistency range of bituminous substances. It is claimed that the temperature range between the liquefying-point and solidifying-point constitutes a numerical index for each bituminous substance, representing its resistance to temperature changes. The higher this index, the more resistant will the substance be, and conversely, the lower this index, the more rapidly will the substance pass from the solid into the liquid state upon being subjected to

increasing temperatures. The indices applicable to typical substances are given in Table CXXXI.

TABLE CXXXI
RELATIONSHIP BETWEEN LIQUEFYING AND SOFTENING-POINTS

	Softening-point		Liquefying-point	Solidifying-point	Temperature Range
	R. and B.	K. and S.			
Montezuma 1.....	66.0° C.	52.0° C.	77.0° C.	- 6.5° C.	83.5° C.
Montezuma 3.....	58.0	43.0	69.0	- 17.6	86.6
Mexpetebano 1.....	65.0	49.5	75.0	- 7.9	82.9
Mexpetebano 3.....	57.5	41.5	68.0	- 14.0	82.0
Mexpetebano 6.....	49.0	34.0	60.0	- 19.4	79.4
Mexpetebano 9.....	43.0	29.0	54.0	- 25.9	79.9
Mexphalt DX.....	66.0	48.5	76.0	- 8.4	84.4
Mexphalt E.....	57.0	42.5	68.5	- 15.7	84.2
Mexphalt E1.....	53.5	37.0	64.0	- 16.8	80.8
Sprazuma 5.....	53.5	38.0	64.5	- 18.8	83.3
Sprazuma 6.....	55.0	38.0	64.5	- 17.1	81.6
Spramex.....	41.0	25.0	51.0	- 28.1	79.1
Trinidad Asphalt...	94.0	77.0	104.0	+ 17.5	86.5
Tar 1.....	3.5	- 6.5	9.0	- 38.0	47.0
Tar 2.....	7.9	+ 0.4	14.0	- 33.6	47.6
Tar 3.....	30.0	21.0	37.5	- 18.7	56.2
Coal-tar Pitch A....	76.0	66.5	83.5	+ 27.2	56.3
Coal-tar Pitch B....	78.5	67.0	86.8	+ 28.7	58.1

It is further claimed that the R. and B. and the K. and S. softening-points represent the temperatures at which the substance has attained a *definite* degree of softness, and that these bear a fixed relation to the range in temperature and/or softness between the liquefying- and the solidifying-points, as expressed by the following equation:

$$\frac{\text{Temp. range between softening-point and solidifying-point}}{\text{Temp. range between liquefying-point and solidifying-point}} = C$$

Where C represents a constant, which has been ascertained experimentally to be:

$$C_r = 0.8721 \text{ for R. and B. method.}$$

$$C_k = 0.6816 \text{ for K. and S. method.}$$

The degree of softness at the liquefying temperature (i.e., liquefying-point) is accordingly fixed at one extreme of an arbitrary scale of softness and assigned a value of 1.0000. Similarly, the degree of softness at the solidifying temperature (i.e., solidifying-point) is fixed at the lower extreme of this scale and assigned a value of 0.0000. Expressed on the same scale, the degree of soft-

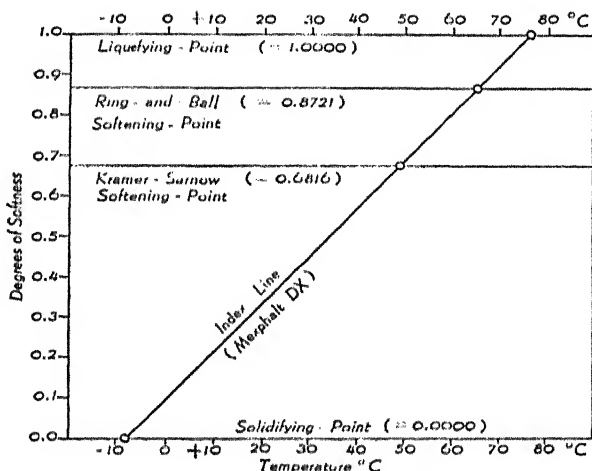


FIG. 271.—Relationship Between the Liquefying-point and the R. and B. and K. and S. Softening-points.

ness at the R. and B. softening temperature will equal 0.8721 and at the K. and S. softening temperature 0.6816. This relationship is illustrated graphically in Fig. 271.

Expressed mathematically, these relationships take the following form:

If L = Liquefying-point temperature (Ubbelohde method)

S = Solidifying-point temperature (Metzger method)

R = Softening-point temperature (R. and B. method)

K = Softening-point temperature (K. and S. method)

$C_r = 0.8721$

$C_k = 0.6816$

Then:

$$\frac{R - S}{L - S} = C_r = 0.8721$$

and

$$R = 0.8721(L - S) + S \quad (1)$$

also

$$\frac{K - S}{L - S} = C_k = 0.6816$$

and

$$K = 0.6816(L - S) + S \quad (2)$$

From (1) and (2) we derive the following:

$$R - 0.9721(L - S) = K - 0.6816(L - S)$$

and

$$R = K + 0.1905(L - S) \quad (3)$$

$$K = R - 0.1905(L - S) \quad (4)$$

From the foregoing, it follows that for any bituminous substance, these relationships hold true:

(a) If the liquefying- and solidifying-points are known, then it is possible to calculate either the R. and B. or the K. and S. softening-point (see formulas 1 and 2).

(b) The R. and B. and K and S. softening-points bear a definite relation to each other, and either one may be computed from the other (see formulas 3 and 4).

These facts have been borne out experimentally with a reasonable degree of accuracy.

Fig. 272 shows the relations between the solidifying-point, the K. and S. softening-point, the R. and B. softening-point, and the liquefying-point of certain of the foregoing substances.

The usefulness of these tests in examining mixtures of bituminous substances will appear from Figs. 273, 274, 275 and 276.

A modification of this test has been proposed by F. Höppler, who devised an instrument for ascertaining the flow-point which at the same time arranges to chart flow-curves. Logarithmic linear relations have been reported between the absolute viscosity, penetration, softening-point and flow-point, and the elastic properties of asphalts have been measured over a range of temperatures.¹⁸⁷

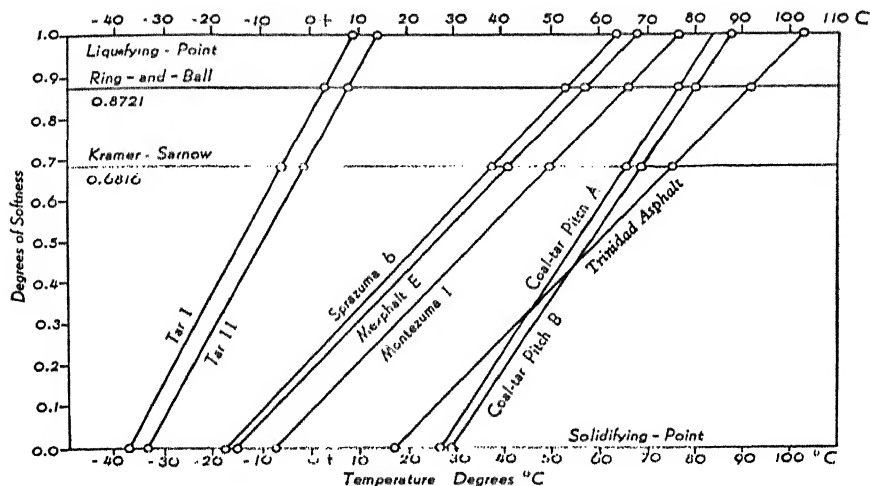


FIG. 272.—Relationship Between the Solidifying-point and the R. and B. and K. and S. Softening-points of Typical Asphalts and Tars.

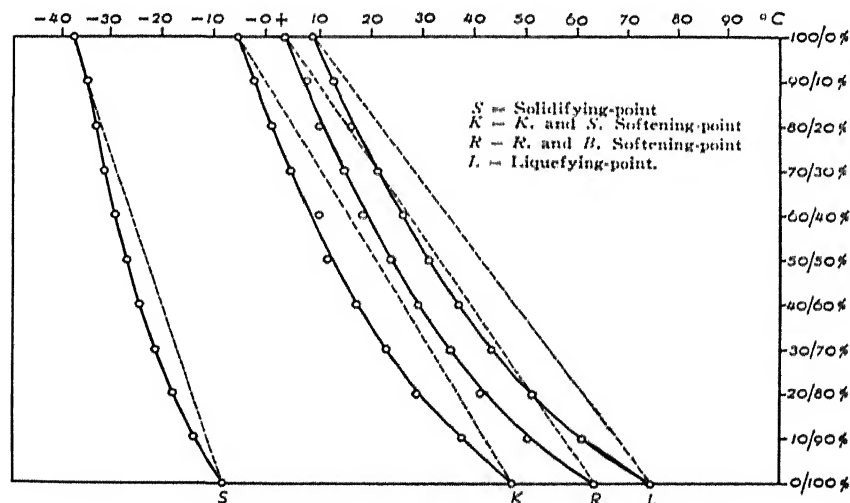


FIG. 273.—Mixtures of Tar I and Mexpetebano.

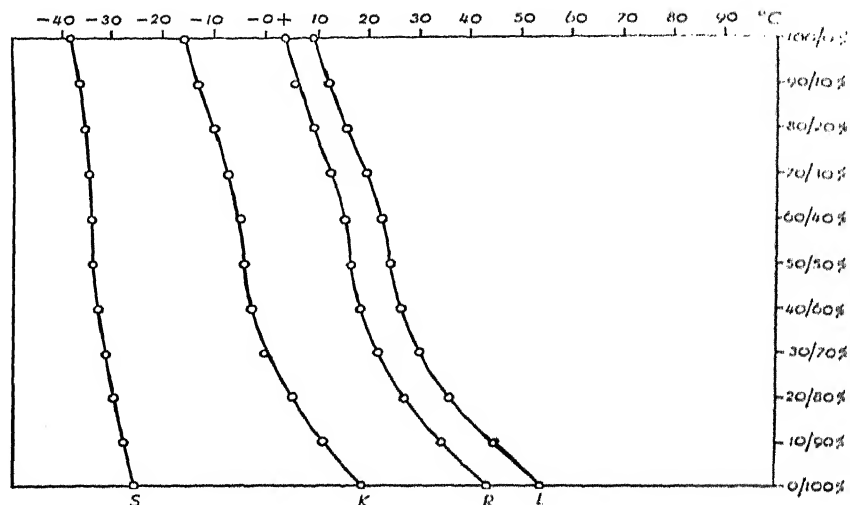


FIG. 274.—Mixtures of Tar 1 and Mexpetebano 9.

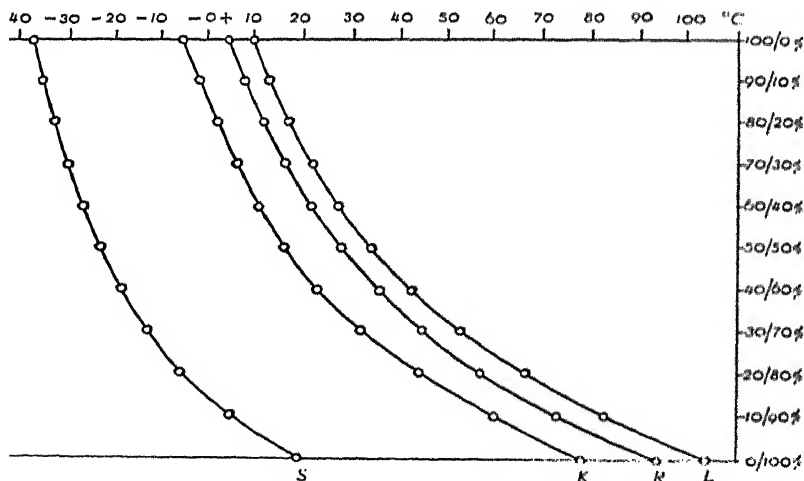


FIG. 275.—Mixtures of Tar 1 and Trinidad Asphalt.

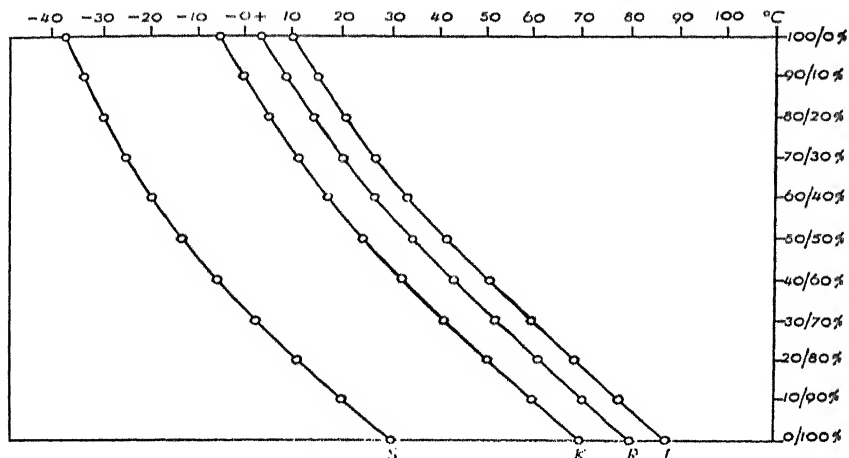


FIG. 276.—Mixtures of Tar 1 and Coal-tar Pitch B.

SOFTENING-POINT OR FUSING-POINT*

This constitutes one of the most valuable all-around tests. It is used for purposes of identification, especially with materials fusing at a high temperature, such as the asphaltites, and is particularly useful in this connection upon taking into consideration the specific gravity and hardness. It is also used for ascertaining the adaptability of a bituminous material for certain definite usages, including its resistance to the sun or artificial heat. The fusing point serves to gauge the uniformity of supply, and on account of its rapidity and accuracy, is used extensively for purposes of factory control. Various methods have been proposed for this purpose, viz.: change in appearance on heating,¹³⁸ heating the powdered substance with sulfuric acid,¹³⁹ slide test,¹⁴⁰ sagging tests,¹⁴¹ dropping tests,¹⁴² extrusion under pressure,¹⁴³ releasing a ball under tension,¹⁴⁴ heating the substance in contact with a thermo-couple and observing the

* The terms "fusing-point" and "softening-point" have been used throughout the text in place of the phrase "melting-point," since the former are more expressive of the behavior of fusible bituminous substances under the influence of heat. They pass *gradually* from the solid to the liquid condition, the transition taking place slowly, owing to the heterogeneous character of the substances present. The phrase "melting-point" is more appropriately applied to chemical substances having a definite composition, which melt sharply, and within a narrow temperature range.

point at which the temperature remains stationary over an appreciable length of time (due to the material absorbing its latent heat of fusion).¹⁴⁵

Test 15a. Krämer-Sarnow Method. This method is rapid, accurate, and adapts itself either to soft or hard bituminous materials, from residual oils up to grahamite. Its range is greater than that of any other fusing-point method.

It was first proposed by G. Krämer and C. Sarnow.¹⁴⁶ Various modifications have been suggested from time to time.¹⁴⁷ The author, after a careful study of this method, recommends the following procedure:¹⁴⁸

I. For Bituminous Substances Fusing below 176° F. This method consists in heating a plug of the bituminous substance 5 mm. long, in an open glass tube, 6–7 mm. internal diameter, and about 8 cm. long, the plug supporting 5 g. mercury, and the tube being immersed in a vessel of water, the level of which reaches approximately the center of the mercury column. In making the test, a thermometer is suspended in the liquid, so that its bulb will be at the same level as the plug of bituminous material. The thermometer is

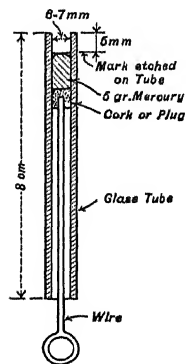


FIG. 277.—Method of Filling K. and S. Fusing-point Tubes.

supported in a separate glass tube of the same thickness and diameter as the other tube, but differing therefrom in having its lower end sealed, and containing sufficient mercury to surround the bulb. The water is heated at a uniform rate of 4° F. per minute, and the temperature at which the mercury drops through the plug of bituminous material recorded as its fusing temperature. The tube containing the bituminous substance may have a mark etched 5 mm. from the end, as a convenient guide for the quantity of bituminous material to be introduced. The plug of bituminous material may be introduced into the tube by inverting it and inserting into its lower end a well-fitting cork or wooden plug fastened to a stiff wire. The mercury is poured on same, and the plug raised or lowered until the meniscus of the mercury coincides with the mark etched on the tube. The bituminous material is then melted at a temperature slightly above its fusing-point and poured on top of the mer-

cury, completely to fill the tube, which should be warmed slightly. When cool, the bituminous material is levelled off even with the end of the tube, whereupon the tube is inverted and the plug withdrawn. This is illustrated in Fig. 277.

The mercury is measured from a heavy-walled capillary tube of 1 mm. bore, terminating in a three-way cock, as illustrated in

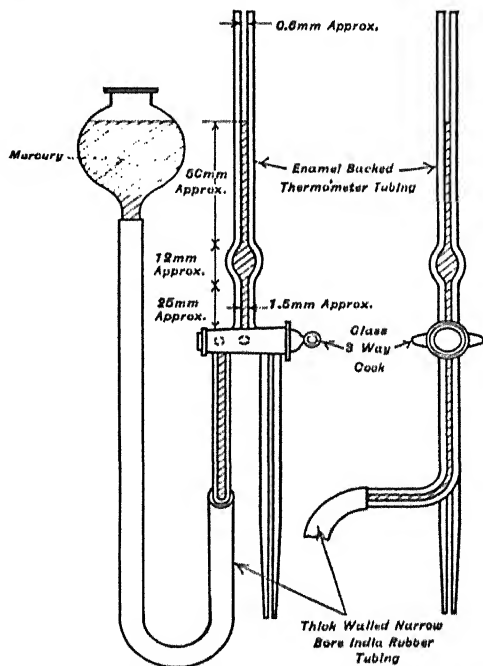


FIG. 278.—Krämer and Sarnow Mercury Pipette.

Fig. 278, and calibrated to hold exactly 5.00 ± 0.5 g. mercury at room temperature. The short limb of the tube is connected with a movable reservoir containing mercury, the height of which is adjusted so the mercury in the capillary tube exactly reaches the graduation.

The heating is conveniently effected by an electrical device described by the author,¹⁴⁹ illustrated in Fig. 279, composed of a coil of resistance wire to be immersed in the liquid bath containing the

fusing-point tubes, and connected with a rheostat, by means of which the temperature may be controlled accurately. Three slabs of slate or asbestos-cement, *a*, *b* and *c*, are fastened together with three small bronze bolts *d*-1, *d*-2 and *d*-3, also three large bolts, *e*-1, *e*-2 and *e*-3 enclosed in glass tubes *f* and *g* respectively, to prevent short-circuiting. The coil *h* consists of 10 yd. of cotton-covered No. 30 Nichrome resistance wire wound in a single layer around the tubes, and connected with the bolts *e*-2 and *e*-3, which in turn terminate in binding-posts *i* and *j*. The coil after being assembled is treated with a high-grade insulating varnish and baked until hard. Ten holes are drilled in the slab *a*, three *e*, for the large bolts, six *k* for the fusing-point tubes and one *l* for the thermometer tube. The coil as described offers a resistance of 75 ohms, and allows a passage of approximately 1.5 amperes at a potential of 110 volts. It will raise the temperature of 500 to 600 ml. of water to the boiling-point in a few minutes, when the full current is applied.

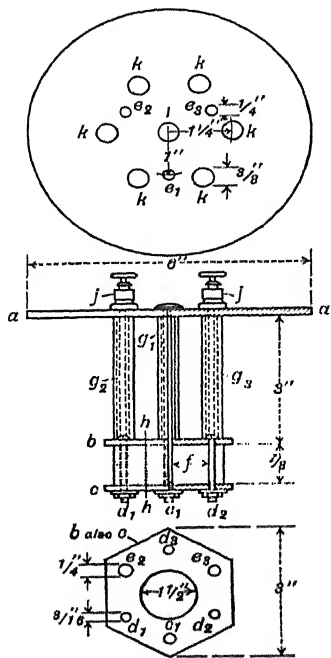


FIG. 279.—Heating Coil for K. and S. Fusing-point Tester.

added 4 drops of concentrated sulfuric acid. The apparatus is assembled as shown in Fig. 280. A *direct* current of 110 volts is used in conjunction with a rheostat provided with 25 to 35 notches, having a carrying capacity ranging from $\frac{1}{2}$ up to 100 amperes (corresponding to a resistance of 220 down to 1.1 ohms).¹⁵⁰

The apparatus is assembled as illustrated in Fig. 281. The heating coil *A* carrying the thermometer *B* and the fusing-point tubes *C* is counterbalanced by the weight *D*, so it may be raised or

lowered into the beaker *E* holding 500–600 ml. of water. The heating coil is connected with a rheostat *F* and a switch *G* in parallel with an 8 c.p. incandescent lamp *H* behind the beaker to illuminate the fusing-point tubes, and a 32 c.p. lamp *I* to light up the interior of the apparatus. *J* represents the mercury measuring-device, and *K* a clock from which the hour hand has been removed, and the

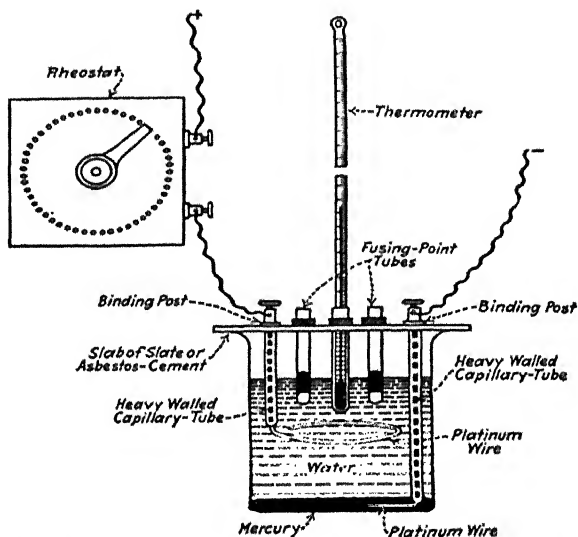


FIG. 280.—Resistance Cell for Fusing-point Determination.

dial graduated in 240 divisions representing degrees Fahrenheit. The rise in temperature is synchronized with the minute hand of the clock and controlled by the rheostat to increase *exactly* $4^{\circ} F.$ *per minute*. The initial temperature of the water should be at least $25^{\circ} F.$ lower than the fusing-point of the material to be examined. Six tests may be run simultaneously.

The heating coil is simple to construct, easy to operate, and insures a perfect temperature control. Owing to its skeleton construction, the heat is rapidly dissipated, and there is no danger of the coil burning out, provided it is kept immersed in the water while the current is on. In the author's laboratory, where the coils are

in daily use, they last from two to three years, and when burnt out the wiring may be renewed in a few minutes' time.

II. For Bituminous Substances Fusing above 176° F. In this case the heating is performed by a direct flame, as illustrated in Fig.

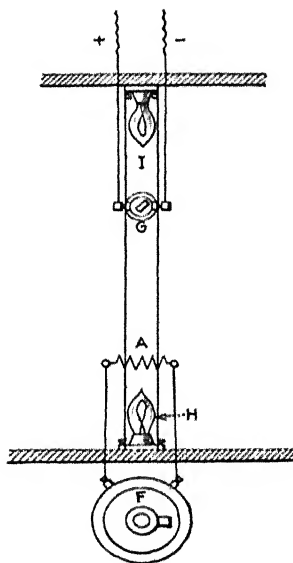
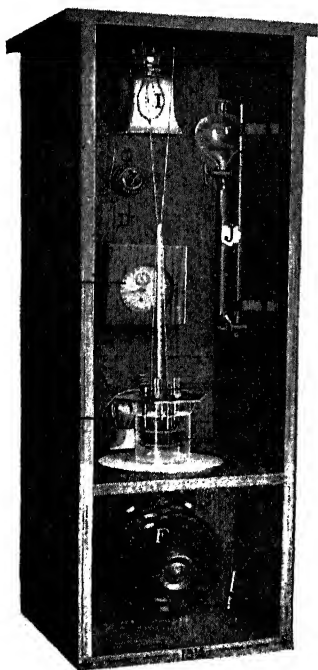


FIG. 281.—K. and S. Fusing-point Tester.

282, the water being replaced with castor oil which may be heated safely to about 600° F. This method may be used for determining the fusing-point of asphaltites, including grahamite. A small quantity of the high fusing-point bituminous material is powdered and compressed in the lower end of the fusing-point tube, whereupon it is carefully heated above the flame of a burner, until the plug of bituminous material softens and fuses to the tube, which is evidenced by the color changing from a dull to a glossy black. The tube is then stood upright against a block of wood, a snug-fitting glass rod

inserted in the upper end, and pressed against the softened bituminous material to compact it into a solid mass 7 to 9 mm. long. On cooling, the plug is then carefully scraped from the lower end of the tube until *exactly* 5 mm. remains, leaving an air space of 2 to 4 mm. between the plug and the lower end of the tube. Care should be taken when suspending the fusing-point tube in the heating bath, to allow the free space below the plug to remain filled with air,

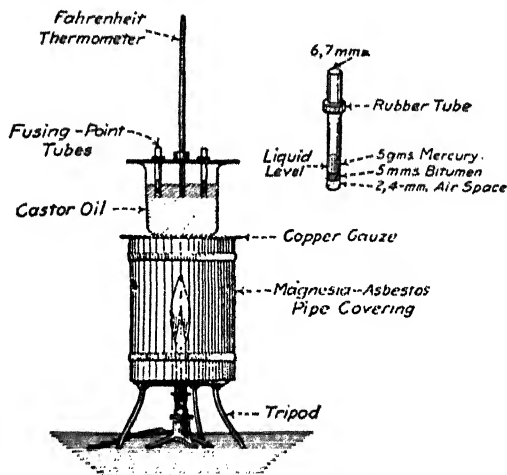


FIG. 282.—K. and S. Tester for High Fusing-point Substances.

otherwise oil will come in contact with and prematurely soften the bituminous material. The bath is heated at the uniform speed of 4° F. per minute.¹⁵¹

This method has been standardized in Great Britain as follows:¹⁵² The apparatus shall be as shown in Fig. 283, and constructed as described below:

The water-bath consists of a 600-ml. beaker and a 250-ml. beaker, both of tall form without spout. The beakers are mounted one within the other and with their upper edges in the same plane; this may conveniently be effected by making a support as follows:

A strip of copper foil, about 12 mm. wide, is placed round the outside of the smaller beaker and is soldered at the overlap to form a collar. The widest beaker available of this form and capacity

(250 ml.) is selected for the purpose, so that it can be replaced readily in the event of breakage. Three other strips of copper foil, also about 12 mm. wide, are soldered to the collar, at right angles to its length and at equal distances apart. A similar collar is prepared for the larger beaker and, with the smaller one turned upside down and the larger one placed concentrically over it, the three strips from the smaller collar are soldered, when pulled tight, to the larger collar.

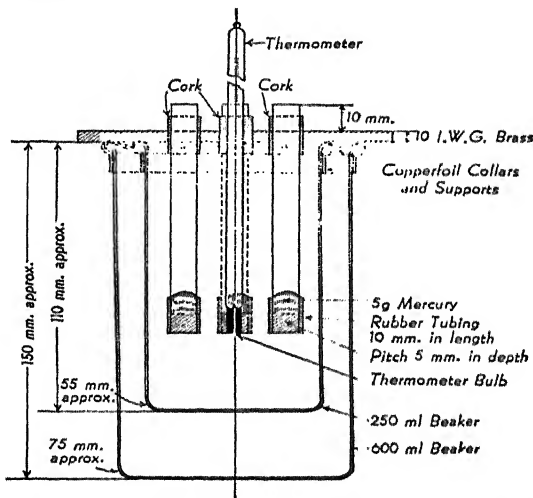


FIG. 283.—Krämer and Sarnow Assembled Apparatus.

The mercury tube support consists of a disc of 10 I.W.G. brass plate, about 100 mm. in diameter, bored with five holes, each 12.5 mm. in diameter and so placed that one is in the exact center and the others about equidistant from it and also from one another. Their distance from the center of the disc is determined by the internal diameter of the inner beaker, their positions being such that the mercury containers which are to pass through them shall be situated as nearly as possible midway between the center and wall of the beaker. It is advisable to number the radial holes 1, 2, 3 and 4 for identification purposes during the test.

The mercury containers consist of glass tubes, 95 mm. in length and $6.5 \text{ mm.} \pm 0.1$ in internal diameter. They shall be about 8 mm. in external diameter and one end of each must be accurately ground at right angles to the axis.

The pitch containers are short lengths of brass or stainless steel

tubing, 6.5 mm. \pm 0.1 in internal diameter. They shall be ground level at their ends, at right angles to their length, which must be 5.0 mm. \pm 0.2. The tubes shall be about 8 mm. in external diameter.

A standard thermometer having the range 0° to 120° C.

A filter paper, cut to the required size and held down by a thin flat metal ring about 6 mm. wide and of such diameter as will allow it just to fit the inner beaker, may be employed to catch the molten pitch and prevent it from contaminating the bottom of the beaker.

The apparatus is assembled on a tripod and is protected by a standard draught screen of sufficient height for the purpose when standing on the bench. A Bunsen burner, with a governor if the gas supply is liable to fluctuate. A pitch melting bath. A mercury pipette (Fig. 278) consisting of a three-way, oblique bore, accurately ground stopcock, having tubes about 1.5 mm. in internal diameter and about 6 mm. in external diameter.

Test 15b. Ring-and-ball Method. This has been standardized as follows: ¹⁵³

The softening of bituminous materials generally takes place at no definite moment or temperature. As the temperature rises, they gradually and imperceptibly change from a brittle or exceedingly thick and slow-flowing material to a softer and less viscous liquid. For this reason the determination of the softening-point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

The apparatus shall consist of the following:

(a) A brass ring 15.875 mm. ($\frac{5}{8}$ in.) in inside diameter and 6.35 mm. ($\frac{1}{4}$ in.) deep; thickness of wall, 2.38 mm. ($\frac{3}{32}$ in.); permissible variation on inside diameter and thickness of ring 0.25 mm. (0.01 in.). This ring shall be attached in a convenient manner to a No. 13 B. & S. gage brass wire (diameter 1.83 mm. = 0.072 in.). See Fig. 284.

(b) A steel ball 9.53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3.45 and 3.55 g.

(c) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, low form, meets this requirement.)

The sample shall be melted at the lowest possible temperature to avoid loss of volatile constituents ¹⁵⁴ and stirred thoroughly,

avoiding incorporating air bubbles in the mass, and then poured into the ring so as to leave an excess on cooling. The ring, while being filled, should rest on a brass plate which has been amalgamated to prevent the bituminous material from adhering to it. After cooling, the excess material shall be cut off cleanly with a slightly heated knife.

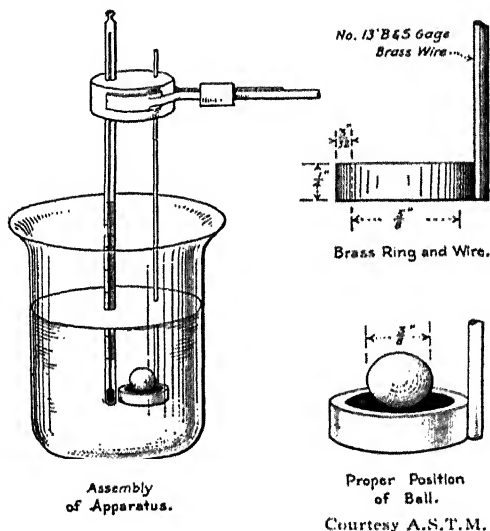


FIG. 284.—Apparatus for Ring-and-ball Method.

I. For Asphalts Fusing at 80° C. (176° F.) or Below. Use a thermometer which shall be graduated in either Centigrade or Fahrenheit degrees as may be specified, the ranges being -2 to $+80^{\circ}$ C. or 30 to 180° F., respectively. Fill the glass vessel to a depth of substantially 8.25 cm. (3.25 in.) with freshly boiled, distilled water at 5° C. (41° F.). Suspend the ring containing the sample in the water so that the lower surface of the filled ring is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper surface is 5.08 cm. (2 in.) below the surface of water. Place the ball in the water but not on the specimen. Suspend the thermometer so that the bottom of the bulb is level with the bottom of the ring and within 0.635 cm. ($\frac{1}{4}$ in.), but not touching the

ring. Maintain the temperature of the water at 5°C . (41°F .) for fifteen minutes.* With suitable forceps, place the ball in the center of the upper surface of the bitumen in the ring, thus completing the assembly as in Fig. 284. Apply the heat in such a manner that the temperature of the water is raised 5°C . (9°F .) each minute. The temperature recorded by the thermometer at the instant the bituminous material touches the bottom of the glass vessel shall be reported as the softening-point. No correction shall be made for emergent stem. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be $\pm 0.5^{\circ}\text{C}$. (0.9°F .). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

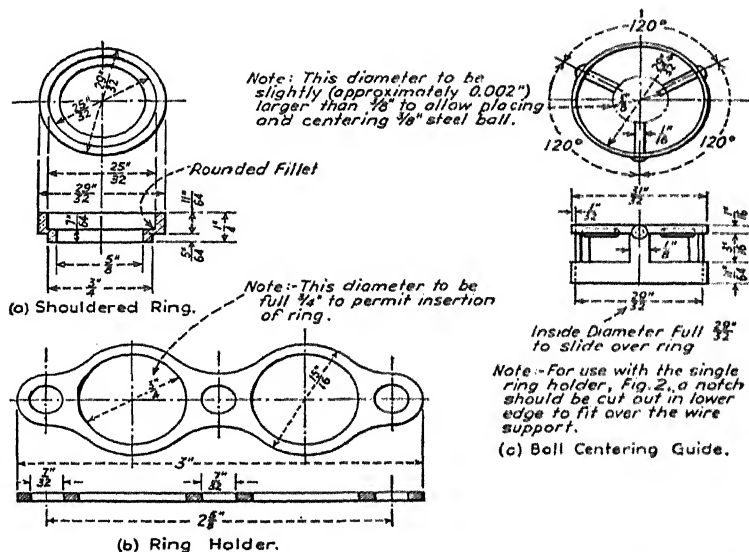
II. For Asphalts Fusing above 80°C . (176°F .). The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 30 to 160°C ., or 85 to 320°F ., respectively. The same method as given above shall be employed, except that U. S. P. glycerin shall be used instead of water, and the starting point of the glycerin bath shall be 32°C . (89.6°F .). The bath shall be brought to this temperature and thoroughly agitated, then the apparatus and specimens shall be placed in the bath which shall be maintained under agitation at the starting temperature for fifteen minutes, after which the assembly shall be completed by placing the ball on the center of the specimen and the test carried on as above. In applying the heat, the ring apparatus shall be placed off the center of the container and the burner placed midway between the center and edge of the beaker away from the specimen.

Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results. A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the bituminous material from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning. The limit of accuracy of the test is $\pm 0.5^{\circ}\text{C}$. (0.9°F .).

*NOTE BY AUTHOR.—It will be found more convenient, and the accuracy of test will in no way be sacrificed, if the initial temperature of the water is maintained 25°F . below the softening point of the substance under examination.

III. For Resins, Waxes, etc. To adapt the method for testing substances which may shrink away from the cylindrical ring, as for example, resins, waxes, etc., the following procedure has been standardized:¹⁵⁵

In general, with materials of this type, softening does not take place at a definite temperature. As the temperature rises, these



Courtesy A.S.T.M.

FIG. 285.—Shouldered Ring, Ring Holder, and Ball-centering Guide.

materials gradually and imperceptibly change from brittle or exceedingly thick and slow-flowing materials to softer and less viscous liquids. For this reason the determination of the softening point must be made by a fixed, arbitrary, and closely defined method if the results obtained are to be comparable.

The apparatus shall consist of the following:

(a) Ring: A brass-shouldered ring conforming to the dimensions (Note) shown in Fig. 285 (a). The ring may be attached, by brazing, to a No. 11 B. & S. gage brass wire (diameter 2.31 mm. = 0.091 in.), (see Fig. 286 (A)). A six-unit ring-cluster is shown in Fig. 286 (B).

(b) Ball: A steel ball, 9.53 mm. ($\frac{3}{8}$ in.) in diameter weighing between 3.45 and 3.55 g.

(c) Ball Guide: A ball guide for centering the balls, constructed of brass, and having the shape and dimensions illustrated in Fig. 285 (c).

(d) Container: A glass vessel, capable of being heated, not less than 8.5 cm. ($3\frac{1}{8}$ in.) in diameter and not less than 10.5 cm. ($4\frac{1}{8}$ in.) in depth from the bottom of the flare. (A 600-ml. low-form Griffin beaker meets this requirement. For use with the ring holder an 800-ml. tall form beaker is convenient.)

(e) Support for Ring and Thermometer: Any convenient method for supporting the ring and thermometer may be used pro-



Courtesy Precision Scientific Co.

FIG. 286.—Shouldered Ring—(A) Ball Retaining Ring; (B) Six Unit Ring Cluster.

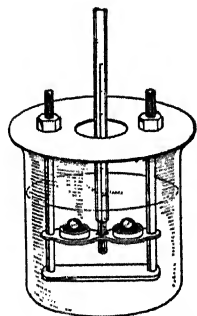
vided it meets the following requirements: The ring shall be supported in a substantially horizontal position. The top of the ring shall be at least 7.6 cm. (3.0 in.) below the top of the container and at least 5.1 cm. (2.0 in.) below the surface of the heating liquid. Using the apparatus in Fig. 285, the bottom of the ring shall be 2.5 cm. (1.0 in.) above the bottom of the glass vessel. If the ring holder is used, the bottom of the ring shall be 2.5 cm. (1.0 in.) above the upper surface of the lower horizontal plate; the lower horizontal plate shall be at least 1.3 cm. ($\frac{1}{2}$ in.) above the bottom of the glass vessel. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom of the ring and within 1.0 cm. ($\frac{3}{8}$ in.) but not touching the ring. For referee work the ring holder shall not carry more than two rings.

(f) Thermometers: (1) An A.S.T.M. Low Softening-point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of -2° to $+80^{\circ}$ C. or $+30^{\circ}$ to $+180^{\circ}$ F. (2) An A.S.T.M. High Softening-point Thermometer, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 30° to 200° C. or 85° to 392° F.

The sample shall be selected from the material to be tested in such a manner as will avoid the inclusion of the surface layer. A

quantity at least twice that necessary to fill the desired number of rings, and in no case less than 40 g., shall be melted immediately in a clean container, using an oven, hot plate, sand or oil bath to prevent local overheating. Care shall be taken to avoid incorporating air bubbles in the sample which shall not be heated above the temperature necessary to pour the material readily without inclusion of air bubbles. The time from the beginning of heating to the pouring of the sample shall not exceed 15 min.

Immediately before filling the rings, they shall be preheated to approximately the temperature at which the material is to be poured. The rings, while being filled, should rest on an amalgamated brass plate. The quantity of material poured into the rings shall be such that after 20-min. cooling at room temperature of materials with softening points below 80° C. (176° F.) and 40-min. cooling for materials of higher softening point, an excess amount will remain which shall then be cut off cleanly with a slightly heated spatula. For routine testing this period of time may be curtailed according to the characteristics of the material under test. In case the test is to be repeated, a clean container and fresh sample shall be used.



Courtesy A.S.T.M.

FIG. 287.—Assembly of Apparatus Showing Two Rings.

The glass container shall be filled to a depth not less than 9 cm. (3.5 in.) with freshly boiled distilled water (or with glycerol for materials melting above 80° C. (176° F.)) which has been cooled to not less than 45° C. (81° F.) below the anticipated softening point, but in no case lower than 5° C. (41° F.) for materials melting below 80° C. (176° F.) and in no case lower than 35° C. (95° F.) for materials melting above 80° C. (176° F.). Provisions shall be made for centering the ball on the upper surface of the sample, either by use of the ball guide or by making a slight indentation in the center of the sample. The latter may be done by pressing the ball or a rounded rod into the material at this point. In the case of hard materials the rod may be heated. The ring containing the sample and ball guide, if used, shall be suspended in the water so that the lower surface of the filled ring is 2.5 cm. (1 in.) above the bottom of the glass container, or 2.5 cm. above the upper surface of the lower horizontal plate which is at least 1.3 cm. (½ in.) above the bottom of the glass vessel (Fig. 287) and so that its upper surface is at least 5.1 cm. (2 in.) below the surface of the water. The ball shall be placed in the water but not on the specimen. The thermometer shall be suspended so that the bottom of

the bulb is level with the bottom of the ring and within 1.0 cm. ($\frac{3}{8}$ in.) but not touching the ring. The initial temperature shall be maintained for 15 min. With suitable forceps the ball shall be placed in the center of the upper surface of the material in the ring.

To facilitate uniform heating, the burner shall be placed midway between the center and the edge of the beaker on a diameter at right angles to the diameter which includes the ring or rings and the thermometer bulb. The effect of drafts on uniformity of heating must be eliminated, using shields if necessary. The heat shall be applied in such manner that the temperature of the bath is raised 5°C. (9°F.) each minute.

Permissible Variation in Rise of Temperature: The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be plus or minus 0.5°C. (0.9°F.). All tests in which the rate of rise exceeds these limits shall be rejected.

The temperature reading to one-half of the smallest scale division indicated by the thermometer at the instant the sample touches the bottom of the container (or the lower horizontal plate) shall be taken as the softening point. No corrections shall be made for emergent stem of the thermometer.

For materials softening around 80°C. (176°F.) the nature of the bath fluid (water or glycerol) shall be reported, since a glycerol bath yields slightly higher results than a water bath.

The use of freshly boiled distilled water is essential, as otherwise air bubbles may form on the specimen and affect the results. Rigid adherence to the prescribed rate of heating is absolutely essential for reproducibility of results.

A thin, amalgamated copper plate or a sheet of filter paper placed on the bottom of the glass vessel will prevent the material from sticking to the bottom of the glass vessel, thereby saving considerable time and trouble in cleaning.

In order to insure uniform heat distribution at all times throughout the bath, a mechanical stirrer should be used.

With care and proper attention to details, duplicate determinations of softening point by this method should not differ by more than 1.0°C. (1.8°F.).

The electrical heating coil described in Test 15a may be used to good advantage in the ring-and-ball method, but the length of nichrome wire should be reduced to 6 yd., to provide for the more rapid heating of the bath, or the resistance-cell illustrated in Fig. 280 may be used as an alternative.

Tests made by the author indicate that the ring-and-ball fusing-points range 15 to 25°F. higher than those obtained by the K. and S. method, although it is questionable whether a fixed mathematical formula adapts itself to the relationship, on account of the basically different fundamentals involved in the two methods of test.¹⁵⁶ Proctor has made a comparison of the K. and S., R. and B., the flowing-temperature and other fusing-point tests.¹⁵⁷

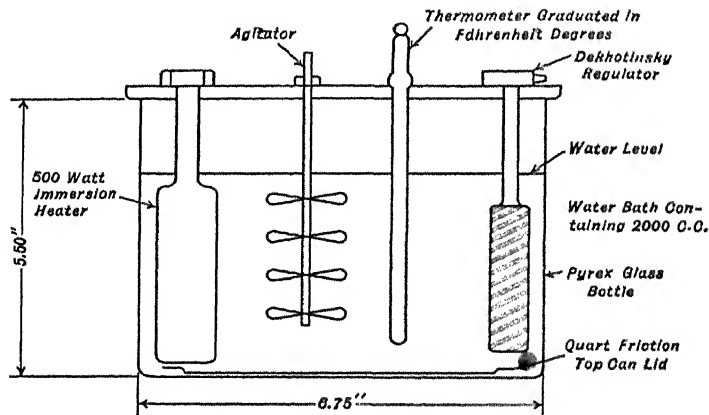


FIG. 288.—Bath for Rapid Method of Test for A.S.T.M. Softening-point.

It has also been observed that the absolute viscosity of petroleum asphalts ranges from 14,000 to 30,000 poises at the ring-and-ball softening-point temperatures.

IV. Rapid Method of Test. A rapid method for ascertaining the A.S.T.M. ring-and-ball softening-point of asphalts, in the range of 90 to 250°C. , in approximately 10 min., has been proposed, as follows:¹⁵⁸ The ring loaded with the ball is immersed in the test bath maintained at a constant temperature, and with a stop-watch the time is recorded for the ball to drop exactly 1 in. and reach the bottom of the bath. Three test baths are recommended, viz.: $130 \pm 0.5^{\circ}\text{F.}$, $180 \pm 0.5^{\circ}\text{F.}$ and $230 \pm 1.0^{\circ}\text{F.}$ Water is used for the first two and glycerin for the 230°F. bath. The baths should be stirred gently in an apparatus as illustrated in Fig. 288. The factors for converting the time in seconds into the A.S.T.M.

TABLE CXXXII

CONVERSION TABLE FOR RAPID METHOD OF TEST FOR A.S.T.M. SOFTENING-POINT

Seconds at 140 F. to A.S.T.M. Softening point		Seconds at 180 F. to A.S.T.M. Softening point		Seconds at 230 F. to A.S.T.M. Softening point	
Reading, sec.	Softening point, deg. Fahr.	Reading, sec.	Softening point deg. Fahr.	Reading, sec.	Softening point, deg. Fahr.
18		21	145	33	180
18.5		21.5	146	34	181
19		22.5	147	34.5	182
20	93	23.5	148	35	183
21	94	24	149	35.5	184
22	95	25	150	36	185
22.5	96	26	151	37	186
23	97	27	152	37.5	187
24	98	28	153	38	188
	99	29	154	39	189
		30	155	39.5	190
27		31.5	156	40.5	191
28		32.5	157	41	192
29		33	158	42	193
30		34.5	159	43	194
31.5		35.5	160	44	195
33		36.5	161	45	196
34.5		38	162	46	197
36		39.5	163	47	198
37		41	164	48	199
39		42.5	165	48.5	200
40.5		44	166	50	201
42		46	167	51	202
44		47.5	168	52	203
46		49	169	53	204
48.5		51	170	54	205
50.5		53	171	55	206
53		55	172	56	207
55		57	173	58	208
56		59.5	174	60	209
61		62	175	61	210
64		63	176	63	211
67		66	177	65	212
70		69.5	178	66	213
74		72	179	69	214
77		75	180	71	215
81		78	181	73	216
85		80.5	182	74	217
90		84	183	78	218
95		86.5	184	81	219
100		90	185	84	220
105		93.5	186	87	221
110		97	187	91	222
118		101	188	95	223
125		105	189	98	224
130		108.5	190	102	225
140		112	191	107	226
150		116.5	192	111	227
159		121	193	117	228
168		125.5	194	122	229
178		130	195	128	230
		133	196	133	231
				140	232
				145	233
				153	234
				160	235
				170	236
				175	237
				185	238
				195	239
				205	240
				215	241
				225	242
				240	243
				250	244
				265	245
				275	246
				290	247
				300	248
				320	249
				340	250
				350	250

softening-point are given in Table CXXXII. The test procedure is summarized in Table CXXXIII.

TABLE CXXXIII

RÉSUMÉ OF RAPID METHOD OF TEST FOR A.S.T.M. SOFTENING-POINT

	For Asphalts in the Range of 90° F. to 140° F. Softening-point.	For Asphalts in the Range of 135° F. to 185° F. Softening-point.	For Asphalts in the Range of 180° F. to 250° F. Softening-point.
1	Place the softening-point rings on the amalgamated Tobin bronze block and pour the liquid asphalt sample into two softening-point rings in a manner to leave an excess on cooling. Pouring temp., 200° to 350° F.		
2	Allow molded samples to remain in air at room temperature exactly 1 min.		
3	Place block and molded samples in 77° F. bath exactly 4 min.	Place block and molded samples in 77° F. bath exactly 4 min.	Place block and molded samples in 115° F. bath exactly 4 min.
4	Remove the block and molded sample from the bath and shave the excess asphalt from the rings with a slightly heated spatula.		
5	Immediately immerse the molds in the 37° F. bath and allow to stand exactly 3 min.	Immediately immerse the molds in the 37° F. bath and allow to stand exactly 3 min.	Immediately immerse the block and molded samples in the 115° F. bath and allow to stand exactly 3 min.
6	Remove the molds from the bath and immediately place the steel balls on the surface of the sample as near to the center of the sample as possible (steel balls at room temperature).		
7	Immediately immerse the molds in the 130° F. bath and start the timer at the same instant.	Immediately immerse the molds in the 180° F. bath and start the timer at the same instant.	Immediately immerse the molds in the 230° F. bath and start the timer at the same instant.
8	Record the times required for the bituminous material to reach the bottom of the bath.		
9	Read estimated A.S.T.M. softening-point from conversion Table CXXXII.		

The following further refinement has been proposed:¹⁵⁹ The shouldered ring-and-ball centering guide should be used, and immersed in a preliminary bath for exactly 5 min. and then immediately in the final testing bath, maintained at the temperatures noted in Table CXXXIV for the various softening-point ranges noted.

TABLE CXXXIV

MODIFIED RAPID METHOD OF TEST FOR A.S.T.M. SOFTENING-POINT

Softening-point Range	Temperature of Preliminary Bath	Temperature of Final Bath
30-45° C. (86-113° F.) 38-50° C. (100-122° F.) 50-65° C. (122-149° F.)	15° C. (60° F.)	{ 35° C. (95° F.) 40° C. (104° F.) 55° C. (131° F.)
65-85° C. (149-185° F.) 75-98° C. (167-208° F.)	25° C. (77° F.)	{ 70° C. (158° F.) 82.5° C. (181° F.)

Graphs are used for converting the time required for the ball to drop 1 in. into the ring-and-ball softening-point, in the form shown in Fig. 289. It is contended that these graphs apply equally to asphalts, coal-tar pitch, water-gas-tar pitch and fatty-acid pitch. Unless the ball falls from the center of the ring and its lower half remains encased in the substance under test, the results will not be reliable.

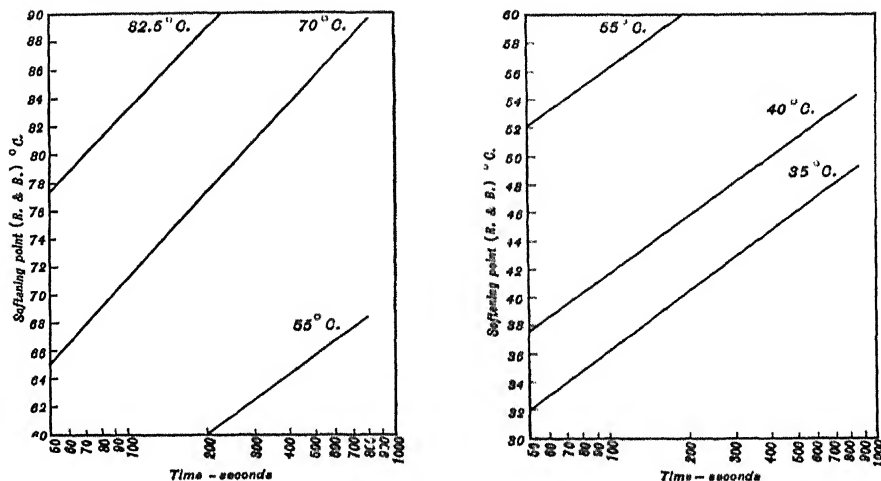


FIG. 289.—Graphs for Modified Rapid Method of Test for A.S.T.M. Softening-point.

Test 15c. Cube Method. This method is restricted to testing tar-pitches. The following procedure has been proposed:¹⁶⁰

The softening of pitch takes place at no definite moment or temperature. As the temperature rises, pitch gradually and imperceptibly changes from a brittle or exceedingly thick and slow-flowing material to a softer and less viscous liquid. For this reason the determination of the softening-point must be made by a fixed, arbitrary and closely defined method if the results obtained are to be comparable.

The methods of test herein described are not applicable to pitches having softening-points above 80° C. (176° F.).

For the purpose of shortening the time required for testing, hard pitches having softening-points between 43° and 80° C.

(109.4° and 176° F.) are cooled at 15.5° C. (60° F.) instead of at 4° C. (39.2° F.) as prescribed for soft pitches.

The apparatus shall consist of the following:

(a) Molds suitable for forming a 12.7-mm. ($\frac{1}{2}$ -in.) cube of pitch. Recommended types are shown in Figs. 290 and 291.

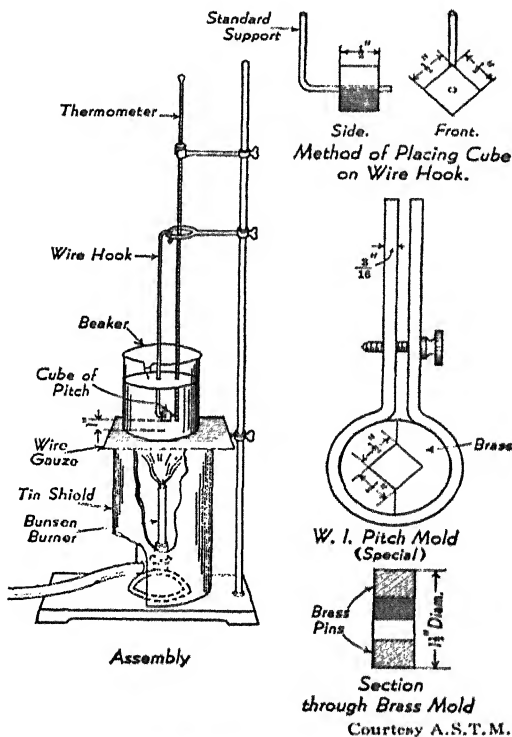


FIG. 290.—Apparatus for Cube-in-water Method.

(b) An L-shaped right-angled hook made of No. 12 B. and S. gage copper wire (diameter 2.05 mm. = 0.0808 in.) the foot of which shall be 2.54 cm. (1 in.) in length. See Fig. 290.

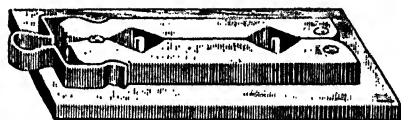
(c) A glass vessel, capable of being heated, not less than 8.5 cm. (3.34 in.) in diameter and measuring 10.5 cm. (4.13 in.) in depth from the bottom of the flare. (A 600-ml. beaker, Griffin low form, meets this requirement.)

(d) A thermometer which shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being -2 to $+80^{\circ}\text{C.}$ or $+30$ to $+180^{\circ}\text{F.}$, respectively.

The pitch shall be formed into a 12.7-mm. ($\frac{1}{2}$ -in.) cube, truly shaped and with sharp edges, either by melting and pouring, or softening and pressing, into a mold. In all cases an excess of pitch shall be used and the surplus material shall be cut off cleanly with a slightly heated knife. The harder pitches specified can ordinarily be molded at room temperature, the softer pitches in water at about



Double Mold



Double Mold with Pins

FIG. 291.—Double Mold for Pitches.

4°C. (39.2°F.). If they are melted, they should first be thoroughly stirred, avoiding incorporating air bubbles in the mass, and then poured into the mold so as to leave an excess on cooling. The mold should rest on a brass plate and the surface of the plate and the interior surfaces of the mold should be amalgamated to prevent the pitch from adhering to them.

I. For Pitches Fusing between 110 and 176°F. The apparatus shall be assembled as shown in Fig. 290. The glass vessel shall be filled to a depth of substantially 9.5 cm. (3.75 in.) with freshly boiled, distilled water at 15.5°C. (60°F.). The cube of pitch shall be placed on the wire as shown in Fig. 290 and suspended in the water so that its lower edge is exactly 2.54 cm. (1 in.) above the bottom of the glass vessel and its upper edge is 5.08 cm. (2 in.) below the surface of the water. The cube shall be allowed to remain in the water for fifteen minutes before applying heat. The thermometer shall be suspended so that the bottom of the bulb is level with the bottom edge of the cube of pitch and within 0.635 cm. ($\frac{1}{4}$ in.), but not touching the cube. The heat shall be applied in such a manner that the temperature of the water is raised 5°C. (9°F.) each minute. The temperature recorded by the thermom-

eter at the instant the pitch touches the bottom of the glass vessel shall be reported as the softening-point. No correction shall be made for emergent stem. The rate of rise of temperature shall be uniform and shall not be averaged over the period of the test. The maximum permissible variation for any minute period after the first three shall be $\pm 0.5^{\circ}$ C. (0.9° F.). All tests in which the rate of rise in temperature exceeds these limits shall be rejected.

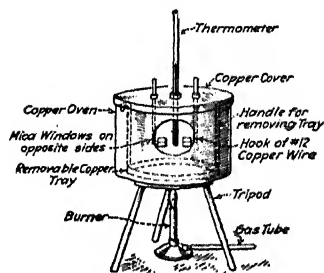


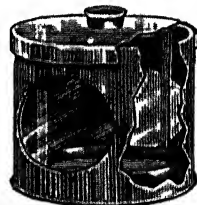
FIG. 292.—Cube-in-air Method for High Fusing-point Substances.

The use of freshly distilled water is essential, as otherwise air bubbles may form on the cube and retard its sinking. Rigid adherence to the prescribed rate of heating is absolutely essential in order to secure accuracy of results. A sheet of paper placed on the bottom of the glass vessel and conveniently weighted will prevent the pitch from sticking to the glass vessel, thereby saving considerable time and trouble in cleaning. The limit of accuracy of the test is $\pm 0.5^{\circ}$ C. (0.9° F.).

III. For Pitches Fusing above 170° F. The heating is performed in an air bath in the apparatus illustrated in Fig. 292. An air-oven attachment is shown in Fig. 293. The cube should be suspended in line with the observation windows, and the thermometer bulb brought to the same level. The temperature is raised 9° F. per minute, and recorded by the thermometer when the cube drops 1 in.

Investigations of the relationship between the cube and the ring-and-ball methods¹⁶¹ indicate that the results vary considerably, depending largely upon the nature of the products tested and their fusing-points. No exact factors can be given. The relation be-

II. For Pitches Fusing below 110° F. Use the same method as given above, except that the water when placed in the glass vessel shall be at a temperature of 4° C. (39.2° F.). The cube shall be allowed to remain fifteen minutes in this water before applying the heat.



Courtesy Precision Scientific Co., Chicago

FIG. 293.—Air-oven Attachment.

tween the fusing-point by the cube method and the results obtained by the Schutte consistency tester, the Engler viscosimeter, also the float test have been investigated.¹⁰²

Figure 294 shows the relation between the softening-points obtained by the cube-in-water and the ring-and-ball methods on three types of tars, each of which were evaporated and samples taken

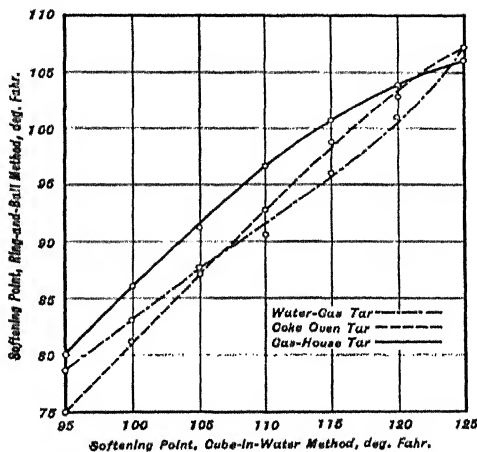


FIG. 294.—Comparison of Ring-and-ball and Cube-in-water Softening-points for Different Types of Tars.

having softening points by the cube-in-water method of 95, 100, 105, 110, 115, 120 and 125° F., respectively.¹⁶³

Test 15d. Compression Method. By this test the extent of softening is measured progressively as the temperature is increased, by a specially constructed instrument illustrated in Fig. 295, known as the Widney Resiliometer.¹⁶⁴ The behavior of the material is expressed graphically and forms a clear picture of the effect of heat on its physical condition.

It consists of a large dial *A* graduated in 100 divisions, each representing .001 in. movement of the plunger *B*, so that one revolution of the pointer, measuring $\frac{1}{8}$ in. in diameter, corresponds to 0.1 in. travel. A small supplemental dial *C* registers the number of revolutions of the pointer on the large dial *A*. By means of a mold, a specimen is prepared measuring exactly 0.1 in. thick and

about $2\frac{1}{2} \times \frac{1}{2}$ in. in area. After allowing the specimen to stand at least twelve hours before testing, it is placed on the base *D*, the plunger *B* brought in contact, and the reading observed on the dial *A*. The catch *E* is then pressed which releases the weight *F* attached to the quadrant *G*. This, by means of the rack *H* and the pinion *I*, transmits the pressure to the plunger *B*. The weight *F* is approximately $2\frac{1}{2}$ lb., so that the material will be subjected to a pressure equivalent to 200 lb. per sq. in. The pressure is maintained until the substance reaches the limit of compression, whereupon the

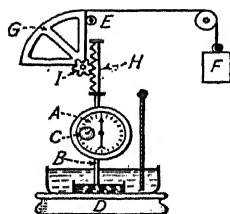


FIG. 295.—Resiliometer for Measuring Compression.

reading on the dial is taken and compared with the original thickness. For example, a specimen showing a thickness originally of 100 mils and giving a reading of 25 mils would be reported to show a compression of 25 per cent. The specimen is heated at the rate of 4° F. per minute and tested every 10° F. at different points over its surface. The diagram in Fig. 296 shows the results obtained upon testing various commercial bituminous substances in the foregoing manner.

Sundry other methods have been suggested to measure the temperature at which a needle or rod under a given load will penetrate the bituminous substance through a predetermined distance; likewise a method involving the measurement of the indentation of a loaded metal sphere acting for a given time at a predetermined temperature.¹⁰⁵

Test 15e. A.S.T.M. Method for Petrolatum. The following procedure has been adopted as standard.¹⁰⁶

The "A.S.T.M. Petrolatum Melting-point" represents the temperature at which petroleum becomes sufficiently fluid to drop from the thermometer used in making the determination under definite prescribed conditions.

The melting-point thermometer shall conform to the specifications for the A.S.T.M. paraffin-wax melting-point thermometer having a range of 38° to 100° C. (or 100° to 180° F.). The bath thermometer shall be of any suitable type, accurate throughout the required range to 2° F.

The test tubes shall be 25 by 100 mm. in size, and shall be sup-

plied with corks grooved at sides to permit air circulation and bored at the exact center to receive the thermometer. A transparent container of not less than 500-ml. capacity that will permit the immersion of the test tube to a depth of at least 75 mm. and still leave a depth of 15 mm. of water below the bottom of the test tube.

Samples of sufficient size that exactly represent the petrolatum under inspection shall be secured. The material shall be melted

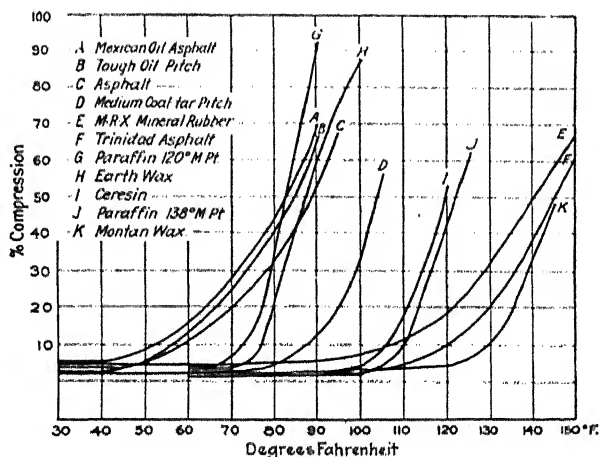


FIG. 296.—Results Obtained with the Resiliometer.

slowly in a casserole or other suitable dish with stirring until the temperature reaches 200° F. A fresh portion of the sample shall be used for each determination. The source of heat shall then be removed and the petrolatum allowed to cool to 15° F. above the temperature that it is anticipated will be its melting-point. The thermometer bulb shall be chilled to 40° F., wiped dry and while still cold thrust into the melted petrolatum so that approximately the lower half is submerged. It shall be withdrawn immediately, held vertically away from the heat until the surface dulls, and then placed for five minutes in a water bath having a temperature not over 60° F.

The thermometer shall be securely fixed in a test tube by means of a cork so that the lowest point is 15 mm. above the bottom of

the test tube. The test tube shall be surrounded with a water bath at a temperature of 60° F. The temperature of the bath shall be raised at a rate of 3° F. per minute to 100° F., then at a rate of 2° F. per minute until the first drop of petrolatum leaves the thermometer. The temperature at this instant shall be recorded.

If the variation of three such determinations does not exceed 2° F., the average of these three determinations shall be reported as the melting-point of the sample under test. If the variation of three determinations is greater than 2° F., two additional determinations shall be made and the average of five determinations reported as the melting-point.

Test 15f. A.S.T.M. Method for Paraffin Wax. The following method has been standardized: ¹⁰⁷

The "Paraffin-Wax Melting-point" represents the temperature at which melted paraffin wax, when allowed to cool under definite prescribed conditions, first shows a minimum rate of temperature change.

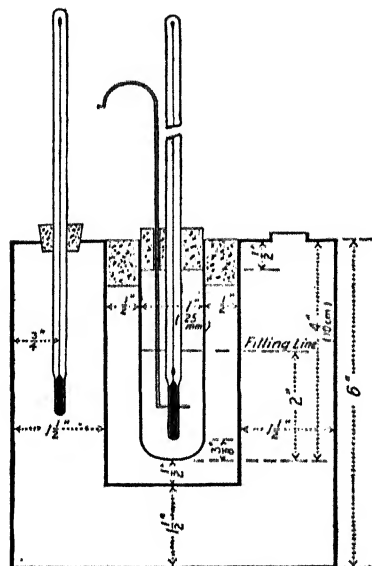
NOTE.—The so-called "American Melting-point" is an arbitrary figure 3° F. higher than the A.S.T.M. Paraffin-Wax Melting-point.

(a) **Wax Container:** Test tube of standard form, 25 mm. (1 in.) outside diameter and 100 mm. (4 in.) long. It may be marked with a filling line, 2 in. above the bottom. This test tube shall be closed in a tightly fitting cork having two openings, one at the center for the melting-point thermometer and the other for a stirrer at one side of the center. The opening for the stirrer may be lined with glass or metal tubing to act as a guide for the stirrer.

(b) **Air Bath:** Suitable water-tight cylinder, 2 in. in inside diameter and 4½ in. deep. This air bath shall be provided with a tightly fitting cork having a central opening for holding the test tube firmly in a vertical position in the center of the air bath.

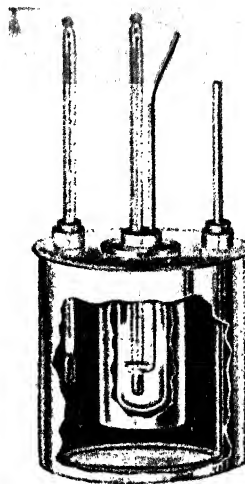
(c) **Water Bath:** Suitable cylinder, 5¼ in. in inside diameter and 6 in. deep. This water bath shall be provided with a suitable cover and with the guides and fasteners necessary to hold the air bath firmly in a vertical central position, so that the sides and bottom of the air bath shall be surrounded by a layer of water 1½ in. in thickness. The water-bath cover shall have a slot for introduction of a suitable stirrer and shall have an opening for the bath

thermometer, so that the latter may be suspended in a vertical position $\frac{3}{4}$ in. from the outside wall of the water bath. Air bath, water bath and water-bath cover may be conveniently made of metal in one assembly as shown in Figs. 297 and 298.



Courtesy A.S.T.M.

FIG. 297.—Apparatus for Determination of Melting Point of Paraffin Wax.



Courtesy Precision Scientific Co.,
Chicago

FIG. 298.—Wax Melting-point Apparatus Assembled.

(d) *Stirrer in Test Tube:* Brass or copper wire, $\frac{1}{8}$ in. in diameter and about 12 in. long. A circular loop, $\frac{1}{2}$ in. in diameter, shall be formed at one end of this wire in such a manner that the loop lies in a horizontal plane when the rest of the wire is in a vertical position. The stirrer thus formed shall be passed through the proper opening in the test-tube cork and the upper end may then be bent into a shape convenient for holding.

(e) *Thermometers:* The Paraffin-Wax Melting-point thermometer shall be a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 38 to

82° C. or 100 to 180° F., respectively. Bath thermometers shall be of any suitable type, accurate to 2° F. throughout the required range.

An average sample of the wax to be tested shall be melted in a suitable container in a water bath whose temperature shall be not more than 35° F. above the approximate melting-point of the wax sample. Direct heat, such as a flame or hot plate, shall not be used and the wax sample shall not be held in the melted condition any longer than necessary.

The test tube shall be filled with melted wax to a height of 2 in. The test-tube cork, carrying the stirrer and the melting-point thermometer with the $3\frac{1}{4}$ -in. immersion line at the under surface of the cork, shall be inserted into the test tube for a distance of $\frac{1}{2}$ in. The lower end of the thermometer bulb shall then be $\frac{3}{8}$ in. from the bottom of the test tube. The air bath being in its proper position in the water bath, the latter shall be filled to within $\frac{1}{2}$ in. of the top with water at a temperature 15 to 20° F. below the approximate melting-point of the wax sample.

The test tube containing the melted wax, with wax stirrer and thermometer in place, shall be inserted into the air bath in a central vertical position, so that the bottom of the test tube is $\frac{1}{2}$ in. from the bottom of the air bath. The temperature of the water bath shall be adjusted by stirring if necessary, so that it shall be lower than the temperature of the wax sample by not more than 30° F. and not less than 25° F., when the wax sample has cooled to a temperature 10° F. above its approximate melting-point.

When these conditions have been obtained, temperature adjustment and stirring of the water bath shall be discontinued. The wax shall be stirred continuously during the remainder of the test, the stirring loop being moved up and down throughout the entire length of the test tube in a steady motion at the rate of 20 complete cycles per minute. The melting-point thermometer reading, estimated to 0.1° F., shall be observed and recorded every thirty seconds. The temperature of the wax will fall gradually at first, will then become almost constant and will then again fall gradually.

The melting-point thermometer reading, estimated to 0.1° F., shall be observed and recorded every thirty seconds, for at least three minutes when the temperature again begins to fall, after re-

maintaining almost constant. The record of temperature reading shall then be inspected and the average of the first four readings that lie within a range of 0.2° F. shall be considered as the uncorrected melting-point. This temperature shall be corrected if necessary for error in the thermometer scale and the corrected temperature shall be reported as the "Paraffin-Wax Melting-point." Duplicate determinations on the same sample should differ by not more than 0.2° F.

A method has been proposed for determining the melting-point of paraffin-wax micro-analytically, where but small quantities are available for test.¹⁶⁸

FLOW-POINT

Test 15g. Richardson's Method. This test¹⁶⁹ consists in forming in a suitable mold, cylinders $\frac{3}{4}$ in. long and $\frac{3}{8}$ in. in diameter of the substance, placing them on a brass plate with corrugations corresponding in size to that of the cylinders, and exposing same at an angle of 45° to the desired temperature in an air-oven for a predetermined length of time. The brass flow-plate is 8 by $2\frac{3}{8}$ in. in size and is provided with four corrugations. The distance which the specimens flow is measured at the conclusion of the test and represents the extent of flow. A modification of this test has been adapted for testing Bituminous Enamel for the inside of steel ships (Chapter XXVII).

LIQUEFYING-POINT

Test 15h. Ubbelohde's Method. This test has been standardized in Germany¹⁷⁰ and represents the temperature at which the substance will fall in the form of a drop under its own weight under prescribed conditions. It has been standardized by the A.S.T.M. as follows for use in determining the dropping-point of lubricating greases:¹⁷¹

Dropping Point. The dropping point is the temperature at which the grease passes from a semisolid to a liquid state under the conditions of the test. It should not be considered as having any bearing upon service performance.

The apparatus shall consist of the following:

(a) Grease Cup: A chromium-plated brass cup conforming to the dimensions shown in Fig. 299.

(b) **Test Tube:** A test tube of heat-resistant glass, with rim, $4 \pm \frac{1}{16}$ in. in length and $1\frac{5}{32} \pm \frac{1}{32}$ in. in inside diameter provided with three indentations about $\frac{3}{4}$ in. from the bottom, equally spaced on the circumference. The depth of these indentations shall be such as to support the grease cup at about the point illustrated in Fig. 299.

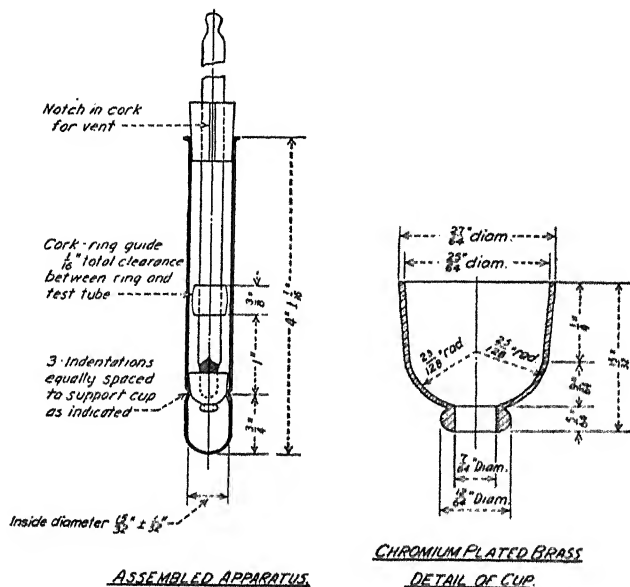


FIG. 299.—Apparatus for Dropping-point Test of Lubricating Grease.

(c) **Thermometers:** Two A.S.T.M. partial-immersion thermometers graduated in either Fahrenheit or Centigrade degrees, as specified, having a range of 20 to 580° F., or -5 to +300° C. and conforming to the requirements for this thermometer as prescribed.

(d) **Accessories:** An oil bath consisting of a 400-ml. beaker and suitable oil, a ring stand and ring for support of the oil bath, clamps for thermometers, two corks as illustrated in Fig. 299, a polished metal rod $\frac{3}{64}$ in. in diameter and 6 in. in length, and suitable means for heating and stirring the oil bath. Heating is preferably done by an immersed electrical-resistance heater regulated by voltage control.

Place the corks on one of the thermometers as illustrated and

adjust the position of the upper cork so that the tip of the thermometer bulb will be about $\frac{1}{8}$ in. above the bottom of the grease cup when the apparatus is assembled for the test. Suspend a second thermometer in the oil bath so that its bulb will be approximately the same level as the bulb of the thermometer in the test tube.

NOTE.—The position of the tip of the thermometer in the test tube is not critical so long as the orifice is not obstructed; with the film of grease applied to the inside of the cup, the thermometer bulb should not be in contact with the grease.

Remove the grease cup and fill by pressing the larger opening into the grease to be tested until the cup is filled, taking care to avoid working of the grease so far as possible. Remove excess grease with a spatula or knife. Gently press the cup, held in a vertical position with the smaller opening at the bottom, down over the metal rod until the latter protrudes about 1 in. Press the rod against the cup in such a manner that the rod makes contact at both the upper and lower peripheries of the cup. Maintain this contact, rotate the cup on the rod along the index finger so as to give it a spiral-like motion down the rod to remove a conical section of the grease which adheres along the rod. When the cup is slipped finally over the end of the rod, a smooth film of reproducible thickness shall be left inside the cup.

Place the cup and the thermometer in the test tube and suspend the test tube in the oil bath with the oil level within $\frac{1}{4}$ in. of the rim. If the cork holding the thermometer in the test tube is properly chosen, the 3-in. immersion mark on the thermometer will coincide with the lower edge of the cork and the assembly should be immersed to this point.

Stir the oil bath and heat at a rate of 8° to 12° F. per min. until the bath reaches a temperature approximately 30° F. below the expected dropping point of the grease. At this point reduce the rate of heating so that the temperature in the test tube will be within 4° F. or less of the temperature in the oil bath before the oil-bath temperature increases an additional 5° F. Continue heating at a rate such that the difference between the temperatures in the test tube and in the oil bath is maintained between 2° and 4° F. This condition is established when the oil bath is heated at a rate of about 2° to 3° F. per min. As the temperature increases, grease will gradually protrude through the orifice of the grease cup. When a drop of grease falls, note the temperatures on the two thermometers and record their average as the dropping point of the grease.

NOTE 1.—Certain greases, for example, some aluminum-base greases, form a drop with a tailing thread upon melting, which may break off or which may hold until the

drop reaches the bottom of the test tube; in any case, the dropping point shall be taken as the temperature when the drop reaches the bottom of the test tube.

NOTE 2.—The dropping points of some greases, particularly those containing aluminum soaps, are known to decrease upon aging, the change being much greater than the deviation permitted in results obtained by different laboratories. Therefore, comparative tests between laboratories should be made within a period of six days.

Two determinations may be made simultaneously in the same bath, provided both samples have approximately the same dropping points.

A sufficient number of determinations shall be made so that the average deviation from the mean is 3° F. or less. The average results so obtained by different operators with different apparatus shall agree within 6° F.

TWISTING-POINT

Test 15i. Taylor's Method. This test has been proposed by H. F. Taylor¹⁷² and is of value in ascertaining the temperature at which the substance may be twisted under torsion without fracturing. The method has been standardized in Great Britain as follows:¹⁷³

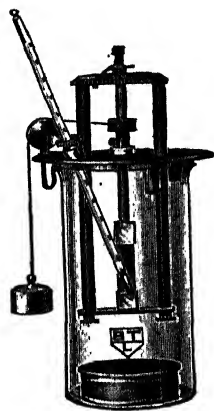


FIG. 300.—Twisting-point Tester.

The testing unit is shown in Figs. 300 and 301. The apparatus is constructed in brass and consists of a device for the application of a standard torque to a square prism of pitch which is being heated at a uniform rate. The torque is exerted by a weight of 150 g. suspended by a thread which, after passing over a pulley, is attached to a drum, the circumference of which is equal in dimension to the perimeter of a cross section of the pitch prism, viz., 51 mm. The drum is fixed to a vertical spindle which is free to rotate. A square socket, 13 mm. x 13 mm. x 15 mm. deep, is fitted to the lower end of the spindle and the upper end of the prism of pitch is fitted into the socket; the lower end rests in a similar socket which is stationary, being attached to the bottom horizontal member of the framework. The distance between the two sockets is exactly 50 mm. The spindle can be raised to permit insertion of the pitch sample. A disc, graduated in degrees of arc, is fixed to

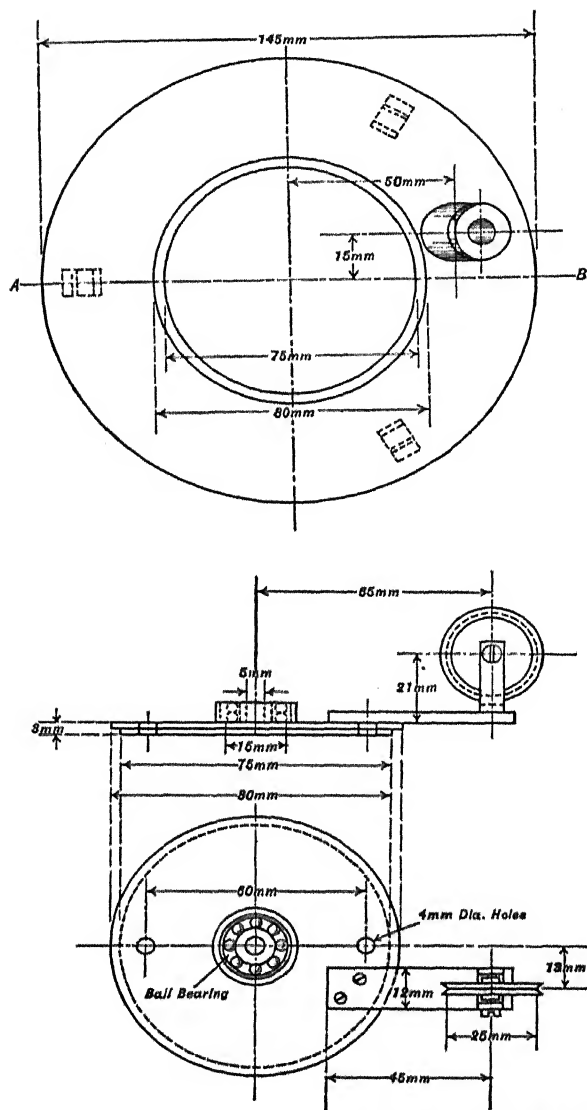


FIG. 301.—Twisting Test for Pitch—Supporting Plate, etc.

the upper horizontal member of the framework and a pointer is fitted at the top of the vertical spindle so that the angle of rotation can be measured. The pointer is so arranged that it can be adjusted to indicate zero at the beginning of each experiment. A small collar supports the vertical spindle on the upper horizontal section of the framework. Ball bearings are provided to ensure free movement; they must be well lubricated (using a *very thin* oil) and maintained in good condition.

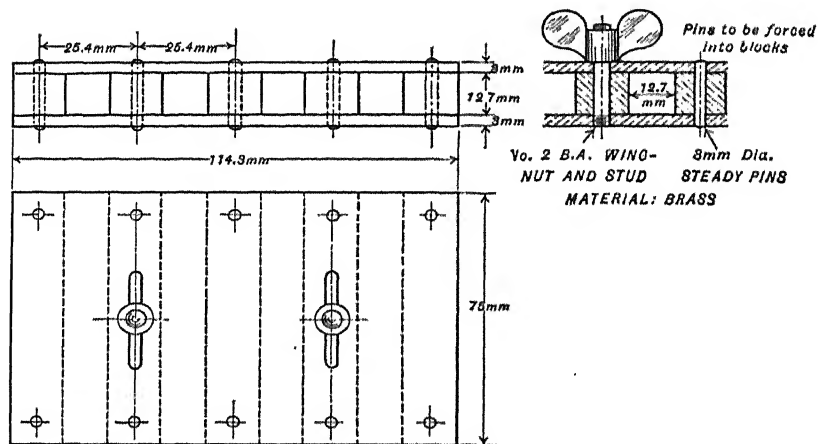


FIG. 302.—Twisting Test for Pitch—Pitch Mold.

The dimensions are approximate, except those shown as 12.7 mm., which must be adhered to within ± 0.05 mm.

A standard thermometer having the range of 0° to 120° C.

A wire gauze disc supported horizontally on a wire gauze cylinder, 25 mm. in height. This serves to break up convection currents.

A heating bath consisting of a 1,500-ml. beaker, of tall form without spout.

A Bunsen burner.

A pitch melting bath consisting of an oil bath made of about 22 gauge copper sheet, supporting a smaller copper vessel in which the pitch is melted; the smaller vessel is fitted with a brass handle.

A brass pitch mold and plate; a suitable form of mold is

shown with the necessary dimensions in Fig. 302, designed to form a half-inch cube.

A portion of the sample, weighing at least 60 g. and powdered if practicable, shall be placed in the inner vessel of the pitch melting bath, the outer vessel of which contains oil at about 80° Centigrade above the expected result of the test. The pitch shall be stirred gently during the heating period to ensure homogeneity and the elimination of air bubbles. As soon as the temperature of the pitch is between 65 and 70° Centigrade above the expected result of the test, the prisms of pitch shall be formed as described below.

The inner surfaces of two of the cavities of the mold and one side of the brass plate shall be amalgamated, the mold assembled and placed with one end resting on the amalgamated side of the brass plate, and the pitch shall be poured into the amalgamated cavities in a thin stream. The quantity of pitch used shall be such that, after ten minutes, a slight excess of pitch remains above the level of the mold. After cooling for 10 min., it is permissible to hasten the subsequent cooling by immersion of the mold under running water. When the mold has cooled to laboratory temperature, the excess of pitch shall be removed by means of a knife and without the application of heat.

If for any reason the test has to be repeated, the pitch melting bath shall be cleaned, a fresh portion of the sample heated and further prisms made as described above.

One of the prisms (75 mm. x 12.7 mm. x 12.7 mm.) shall be fitted into the two brass sockets of the Frankland Taylor machine. It is permissible to facilitate the subsequent removal of the prism from the sockets by rubbing chalk over the surfaces of the pitch prism which will come into contact with the sockets, i.e., over the ends of the pitch prism and along its length for a distance of 15 mm. from each end.

About 1,450 ml. of water at about 20° C. shall be measured into the beaker and the gauze disc and cylinder shall be introduced. The pitch testing unit shall be placed in position as shown in Fig. 300; the level of the water shall be adjusted if necessary by the addition of more water, e.g., from a pipette, so that it is at least 25 mm. above the junction of the vertical spindle and the upper of the

two sockets carrying the pitch prism. The thermometer shall be fitted as shown.

The apparatus shall be allowed to stand for 15 min. in order that the pitch may attain the temperature of the surrounding water. Heat shall then be applied in such a manner that the temperature is raised 2 Centigrade degrees in each minute; this rate shall be maintained within the limits 30 seconds \pm 5 for each Centigrade degree rise after at least 20° Centigrade below the expected result and shall not merely be the average over the period of the test. Rigid adherence to the specified rate is essential and all tests in which the rate in any half minute period is outside the limits shall be rejected.

When the temperature commences to rise, the torsion, which shall not have been applied prior to this stage, shall be imposed by allowing the weight to hang freely and vertically. The pointer shall immediately be set to the zero mark on the scale. The temperature at which the pointer indicates 180° rotation shall be reported.

It is advisable to carry out duplicate tests and in consequence, provision is made for the preparation of a second pitch prism. If duplicate readings for the same sample differ, they shall each be reported as well as the mean.

This instrument may also be used for determining the resilience of the material, as follows: the test is repeated on another specimen and the water maintained at the temperature of the "twist-point" for 20 min. before any weight is attached. The pointer is set at zero, and a 500-g. weight suspended. When the specimen has twisted through an arc of exactly 360 deg., the weight is suddenly lifted by hand and the recoil noted. When equilibrium has been reached, the reading is taken, and the number of degrees' recoil is recorded as the "resilience" or "elasticity" of the material.

For ascertaining the absolute viscosity of the substance, the sample is permitted to twist through two complete revolutions before readings are taken, after which the next two or three complete revolutions are timed with a stop watch and the average time per revolution noted at any predetermined temperature. The viscosity may be calculated from the following formula:

$$V = \frac{mgrlt}{1000\pi^2 R^4}$$

where V = viscosity in kilopoises,
 m = weight of torque (i.e., 50 g.),
 g = acceleration due to gravity (981 g./sec.²),
 r = radius of torque-drum plus cord,
 l = length of sample in mm.,
 t = time of revolution in seconds, and
 R = radius of sample.

A similar method has been proposed for testing the extension and recovery of strips of asphalt to measure its "elastic extension,"

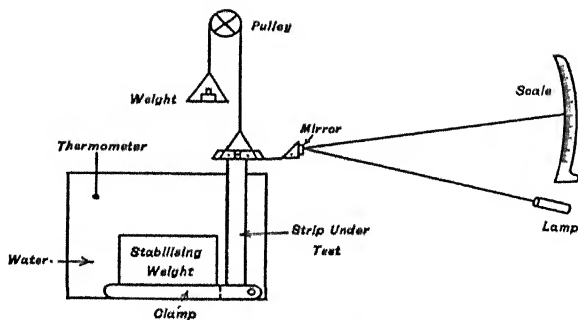


FIG. 303.—Apparatus for Measuring Elastic Extension.

as follows:¹⁷⁴ strips of the material are prepared, 25 cm. long, 2.5 cm. wide, and in thicknesses from 0.2 to 0.6 cm., the lower and upper ends of which are gripped in clamps, as illustrated in Fig. 303. The strip is immersed in a water-bath at 15° C., and subjected to a weight of 60 g. A spot of light from a lamp is reflected by a mirror on a vertical circular scale and the position of the spot is read every 15 seconds. At the end of 2 min., the weight is removed and readings taken of the recovery. The test is repeated at intervals of 10 min. After the first test, the values for extension and recovery fall, until they become constant, after about the third test, whereupon the readings are finally noted, and the index calculated from the following formula:

Let y represent the scale reading (in cm.), r the distance from

axle of prism to grip (2.75 cm.), R the distance from axle of prism to scale (150 cm.), and l the initial length of strip (25 cm.). Then the strain (σ) is given by: $\sigma = ry/2Rl$. For the particular apparatus employed: $\sigma = 3.63 \times 10^{-4}y$. A typical curve is illustrated in Fig. 304.

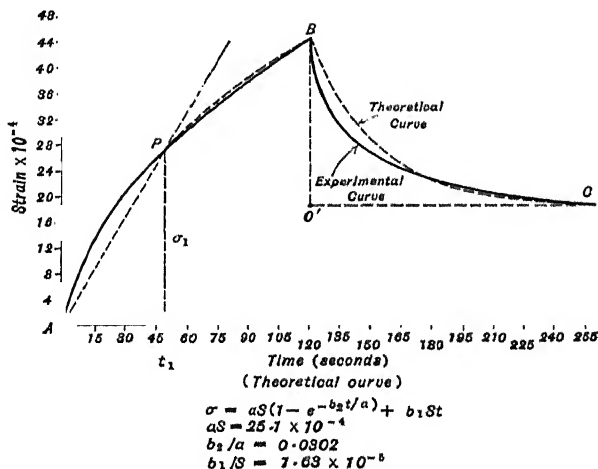


FIG. 304.—Typical Elastic Extension and Recovery Curve.

VOLATILE MATTER

This test is used for identifying various bituminous materials. Thus in the case of asphalts, the volatilization test will often serve to identify soft native asphalts, which contain larger percentages of volatile matter than soft residual or blown petroleum asphalts. Cut-back products also carry a large percentage of volatile constituents.

The test may also be used to determine the adaptability of a bituminous substance for certain definite purposes, where it becomes necessary to heat it to high temperatures, as for example in the paving industry or in manufacturing bituminized roofings and floorings. It likewise serves as a valuable adjunct for gauging the uniformity of supply and for purposes of factory control. It also furnishes an *indication* of the weather-proof properties of the material. Other things being equal, bituminous substances showing the small-

est percentage of volatile matter will prove most weatherproof on exposure to the elements. It should be noted, however, that the volatility test alone must not be taken as the final criterion as to whether or not a bituminous substance is weatherproof, since other factors should also be taken into consideration. The volatility test may be regarded as an accelerated test, showing the loss of volatile constituents exclusive of water which will take place upon exposure to the weather in a relatively thin layer, for a long time.

Test 16. A.S.T.M. Method. The following method has been adopted as standard.¹⁷⁰

This test covers the determination of the loss in weight (exclusive of water) of oil and asphaltic compounds when heated as hereinafter prescribed. The material under examination shall, therefore, first be tested for water and if water is found to be present, it shall be removed by suitable methods of dehydration before the material is subjected to the loss on heating test; or another sample shall be obtained which is free from water.

The oven shall be rectangular in form with double walls and heated by electricity, as illustrated in Fig. 305. Its interior dimensions shall be as follows: height, exclusive of space occupied by the heating element, not less than 29.21 cm. (11.5 in.); width and depth, not less than 29.85 cm. (11.75 in.). The oven shall have in front a tightly fitting hinged door, which shall provide a clear opening substantially of the interior height and width of the oven. The door may contain a window with linear dimensions of at least 10 cm. (4 in.), and with two sheets of glass separated by an air space, through which a vertical thermometer located as specified may be read without opening the door; or the oven may be provided with an inner glass door, through which the thermometer may be observed on opening the outer door momentarily.

The oven shall be adequately ventilated by convection currents of air, and for this purpose the oven shall be provided with openings for the entrance of air and the exit of heated air and vapors. Openings for the entrance of air in interior walls of the oven shall be symmetrically arranged in the bottom or in side walls near the bottom, and shall be so placed that incoming air will circulate around the heating elements; the openings shall have a total area of not less than 1.3 sq. cm. (0.2 sq. in.). Openings for the exit

of heated air and vapors in interior walls of the oven shall be symmetrically arranged in the top or in side walls near the top, and shall have a total area of not less than 1.3 sq. cm. (0.2 sq. in.) nor more than 12.9 sq. cm. (2.0 sq. in.).

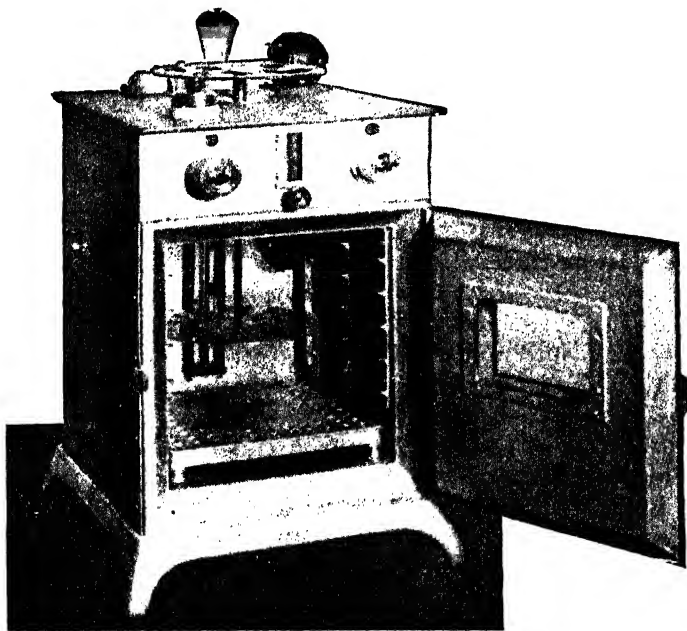


FIG. 305.—Electrically Heated Volatility Oven.

The oven shall be provided with a perforated metal circular shelf approximately 24.77 cm. (9.75 in.) in diameter. A recommended form of aluminum shelf is shown in Fig. 306. This shelf shall be placed in the center of the oven with respect to all dimensions of the interior of the oven, shall be suspended by a vertical shaft, and shall be provided with mechanical means for rotating it at the rate of 5 to 6 r.p.m.

An A.S.T.M. loss-on-heat thermometer graduated in Centigrade degrees, having a range of 155° to 170° C., and conforming to the requirements for this thermometer shall be used.

ference of the circular shelf, in one of the recesses if the recommended shelf is used. The oven shall then be closed and the shelf rotated during the entire test at a rate of 5 to 6 r.p.m. The temperature shall be maintained at $163^{\circ} \pm 1^{\circ} \text{C.}$ ($325^{\circ} \pm 1.8^{\circ} \text{F.}$) for 5 hr. after the sample has been introduced and the oven has again reached that temperature. The 5-hr. period shall start when the temperature reaches 162°C. , and in no case shall the total time that a sample is in the oven be more than 5 hr. and 15 min. At the conclusion of the heating period the sample shall be removed from the oven, cooled to room temperature, weighed to the nearest 0.01 g., and the loss due to heating calculated.

Temperatures shall be determined by means of the specified thermometer, which shall be supported from the shaft of the circular shelf in a vertical position approximately 1.9 cm. (0.75 in.) inside the periphery of the shelf, and with the bottom of the thermometer bulb 0.25 in. above the shelf.

NOTE 1.—If additional periods of heating are desired it is recommended that they be made in successive increments of 5 hr. each.

NOTE 2.—When it is required that the penetration or other characteristics of the sample after heating be determined, the residue should be melted in the container at the lowest possible temperature and thoroughly mixed by stirring, taking care to avoid incorporating air bubbles in the material. For the penetration test, the well-mixed residue shall be brought to standard temperature and tested as prescribed in A.S.T.M. Designation: D 5. For other tests, the well-mixed residue should be tested according to standard conditions as required by the test procedure involved.

Under ordinary circumstances a number of samples having about the same degree of volatility may be tested at the same time. Samples varying greatly in volatility should be tested separately. Where extreme accuracy is required, not more than one material should be tested at one time and duplicate samples of it should be placed simultaneously in the oven. Such duplicates shall check within the limits of accuracy given below. Results obtained on samples showing evidences of foaming during the test shall be rejected.

Up to 5 per cent loss in weight, the results obtained may be considered as correct within 0.5. Above 5 per cent loss in weight the numerical limit of error increases 0.01 for every 0.5 per cent increase in loss by volatilization, as follows:

Volatilization Loss, Per Cent	Numerical Correction	True Volatilization Loss, Per Cent
5.0	±0.50	4.50- 5.50
5.5	±0.51	4.91- 6.01
6.0	±0.52	5.48- 6.52
10.0	±0.60	9.40-10.60
15.0	±0.70	14.30-15.70
25.0	±0.90	24.10-25.90
40.0	±1.20	38.80-41.20

An extension of this test recommended by the author consists in heating the sample to 500° F. for 5 hours. This is advisable in examining relatively non-volatile asphaltic products, which would show but a fraction of a per cent loss by the foregoing method.

A modification of the test consists in heating a thin film, $\frac{1}{8}$ in. thick, to 163° C. (325° F.) for 5 hours and testing the residue for penetration, softening-point and ductility.¹⁷⁶

In Germany, this test is performed in a container 128 mm. in diameter (128 sq. cm. surface area) and 1.5 cm. in depth (internal). Other than this, the remaining details of procedure are the same as the foregoing.¹⁷⁷

Some indication as to the probable performance of road asphalts may be obtained by first heating the specimen to 163° C. (325° F.) for 5 hours in a standard oven, after which the penetration at 77° F. is taken; whereupon the operation is repeated. The logs of the penetration after 5 and 10 hours are plotted against the time in hours. A straight line is then drawn through the two points and extended to 10 penetration, which is used as the "x" axis. The intercept on this "x" axis is termed the "resistance to hardening," which may be calculated from the following equation:¹⁷⁸

$$\text{Resistance to hardening} = 5 \left(\frac{y_1 - 1}{y_1 - y_2} \right) - 5$$

where y_1 = log penetration at 77° F., after 5 hours heating.

y_2 = log penetration at 77° F., after 10 hours heating.

EVAPORATION TEST

Test 16a. A.S.T.M. Method. This test is used principally for testing road oils, in determining the so-called asphalt content, and is carried out by evaporating the specimen under carefully controlled conditions until the residue shows a penetration of exactly 100 at 77° F. (100 g., 5 sec.). The percentage by weight of residue is recorded and furnishes an indication of the quantity of constituents present which may be depended upon to contribute to the durability of the pavement. It will serve to differentiate between straight-distilled and cut-back products. This test has been standardized as follows: ¹⁷⁰

This method of test covers the determination of percentage of residue having a specified penetration at 100 g., 5 seconds, 25° C. (77° F.), obtained by heating a road oil or a semi-solid asphalt having a penetration of more than 100, at a temperature of 249 to 260° C. (480 to 500° F.). When the penetration of the residue is not otherwise stated, it shall be understood to be 100. The residue obtained is available for testing as desired.

The apparatus shall consist of a container, heating bath, hot plate, and thermometer, with necessary accessory apparatus. The container in which the sample is to be tested shall be a flat-bottom, cylindrical seamless tin box, 70 mm. (2¾ in.) in diameter and 45 mm. (1¾ in.) in depth.

NOTE.—The American Can Co.'s 6-oz. Gill style flat-bottom, seamless ointment box, deep pattern, fulfills these requirements.

(a) The heating bath shall be a cast-iron air bath permitting the immersion of the container to a depth of 1¼ in. through an opening ¼ in. larger in diameter than the container. It shall support the container ¼ in. above the hot plate and with at least ¼ in. free air space between the sides of the container and of the air bath below the opening. A suitable air bath is shown in Fig. 307.

(b) The air bath shall be heated upon a suitably mounted hot plate, heated either electrically or by means of a gas flame. The plate shall be capable of maintaining the sample continuously at the required temperature, and apparatus necessary to fulfill this requirement, such as a rheostat or gas pressure regulator, shall be provided.

The thermometer shall be a special thermometer graduated to either Centigrade or Fahrenheit degrees as specified, the ranges being -6 to 400°C. , or $+20$ to $+760^{\circ}\text{F.}$, respectively. The sample as received shall be thoroughly stirred and agitated, to insure a complete mixture before the portion for testing is removed.

One hundred grams ($100.00 \pm 0.10 \text{ g.}$) of the material to be tested shall be weighed into a tared container, which shall then be

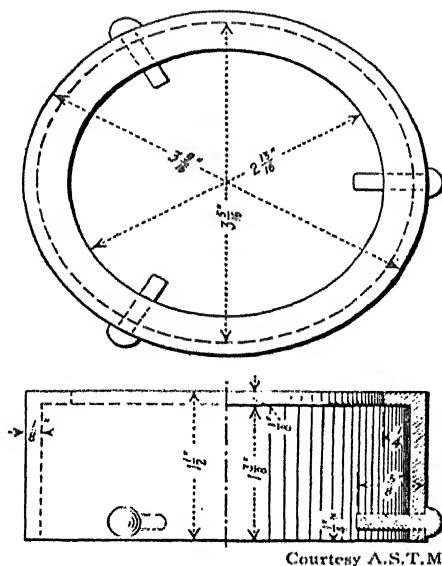


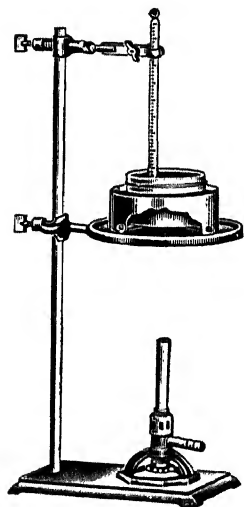
FIG. 307.—Cast-iron Air Bath.

placed in the air-bath in position to be heated. The thermometer shall be supported in the sample equidistant from the sides of the container and with the bottom of the bulb neither more than $\frac{1}{4}$ in. above nor touching the bottom of the container. The bulb shall be completely immersed in the sample throughout the heating. An assembly of the apparatus is shown in Fig. 308.

The sample should be heated as rapidly as possible, to prevent foaming, to a temperature of 249°C. (480°F.) and during the evaporation, the temperature shall be maintained between 249°C. (480°F.) and 260°C. (500°F.). The sample shall be stirred

with the thermometer from time to time to prevent local overheating and, to maintain a homogeneous sample, all cakes of hardened bitumen which form at the sides of the container shall be fluxed in the sample.

An experienced operator can judge approximately what percentage of residue he should obtain to secure the desired penetration. When it is supposed that the residue will show the required penetration, the bitumen on the thermometer which may be readily scraped off shall be returned to the container, which then shall be removed from the air-bath, cooled and weighed. The penetration of the residue shall then be determined in accordance with Test 9*b*, with the exception that the 6-oz. container specified, in which the evaporation has been conducted, shall be used instead of the 3-oz. container specified in Test 9*b*.



Courtesy Precision
Scientific Co.,
Chicago

FIG. 308.—Assembly of Apparatus for Evaporation Test.

It frequently is necessary to make several trials before a residue of the required penetration is obtained. If it is determined to be greater than that required, all water shall be removed from the container and the surface of the sample, and the heating and determination of penetration may be repeated as before. Ordinarily a residue shall be considered as satisfactorily obtained when its penetration is within 15 of that desired, and its percentage by weight of the original sample shall be calculated. When it is necessary to determine more precisely the percentage of residue having the specified penetration, such a percentage shall be computed by interpolation between percentages of two residues, one having a penetration greater and one having a penetration lower than that specified. The percentage shall be reported as:

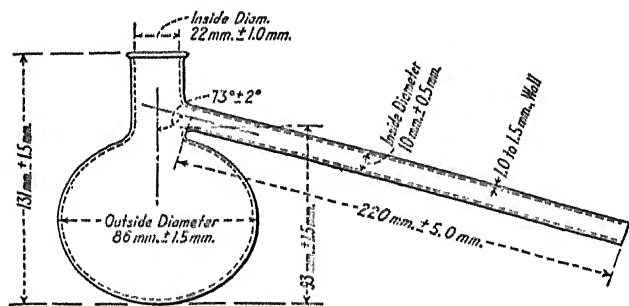
Percentage of residue of penetration (determined) stating, first, the specified penetration, and second, the penetration actually determined for the sample tested or calculated by interpolation.

Certain types of road oil will readily form rings of hard asphalt at the side of the container. Great care should be taken that this material be completely fluxed in the sample before the penetration of the residue is determined.

Duplicate determinations should not differ by more than 1.0 per cent with the same operator nor more than 2.5 per cent between different laboratories.

DISTILLATION TEST

The value of this test is to ascertain the adaptability of bituminous materials for a given use, generally for road treatment; also



Courtesy A.S.T.M.

FIG. 309.—Distillation Flask.

for gauging the uniformity of supply, for purposes of factory control, and most important of all, as a criterion of the quality.

According to Sharples¹⁸⁰ the distillation test as applied to tars becomes of value in identifying the kind used (upon determining the specific gravity of the fractions distilled), as a means of distinguishing a cut-back tar from a straight-distilled tar (upon determining the specific gravity of the fractions, their viscosity, also the fusing-point of the residue), and for detecting the presence of abnormal amounts of naphthalene.

Test 16b. For Tar Products. This test has been standardized as follows:¹⁸¹ The apparatus consists of a flask, condenser tube, shield, receivers and thermometers as specified.

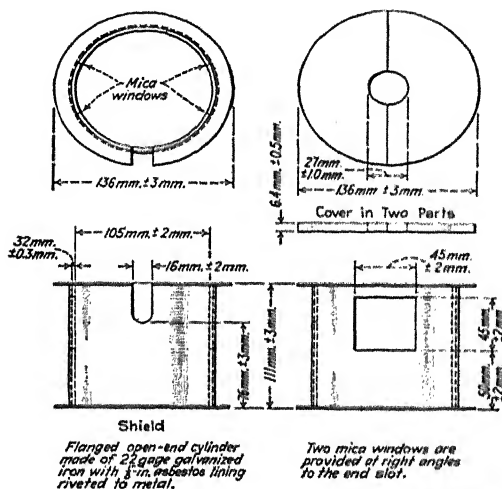
(a) **Flask:** The distillation flask, Fig. 309, shall be a side neck distilling flask, having the following dimensions:

Diameter of bulb, outside.....	86 mm. \pm 1.5 mm.
Diameter of neck, inside.....	22 mm. \pm 1.0 mm.
Diameter of tubulature, inside.....	10.0 mm. \pm 0.5 mm.
Height of flask, outside.....	131 mm. \pm 1.5 mm.
Vertical distance bottom of bulb, outside, to horizontal tangent at tubulature inside.....	93 mm. \pm 1.5 mm.
Length of tubulature.....	220 mm. \pm 5.0 mm.
Angle of tubulature.....	7.3 deg. \pm 2 deg.
Thickness of tubulature wall.....	1.0 to 1.5 mm.

(b) Condenser Tube: The condenser tube shall be a suitable form of tapered glass tubing of the following dimensions:

Outside diameter of small end.....	12.5 mm. \pm 1.5 mm.
Outside diameter of large end.....	28.5 mm. \pm 3.0 mm.
Length.....	360.0 mm. \pm 4.0 mm.
Length of tapered part.....	100.0 mm. \pm 5.0 mm.

(c) Shield: A galvanized iron shield, lined with $\frac{1}{8}$ -in. asbestos, of the form and dimensions shown in Fig. 310 shall be used to pro-



Courtesy A.S.T.M.

FIG. 310.—Shield.

tect the flask from air currents and to prevent radiation. The cover (top) may be of transite board made in two parts, or it may be of galvanized iron lined with $\frac{1}{8}$ -in. asbestos.

(d) Receiver: The distillates shall be collected in tared Erlenmeyer flasks having a capacity of 50 to 100 ml.

(c) *Thermometer:* The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the range being from 0 to 400° C. or 30 to 760° F., respectively.

The sample as received shall be thoroughly stirred and agitated, warming if necessary, to insure a complete mixture before the portion for analysis is removed.

The material may be tested for distillation without dehydration, if water is present not to exceed 2.0 per cent. If water is present

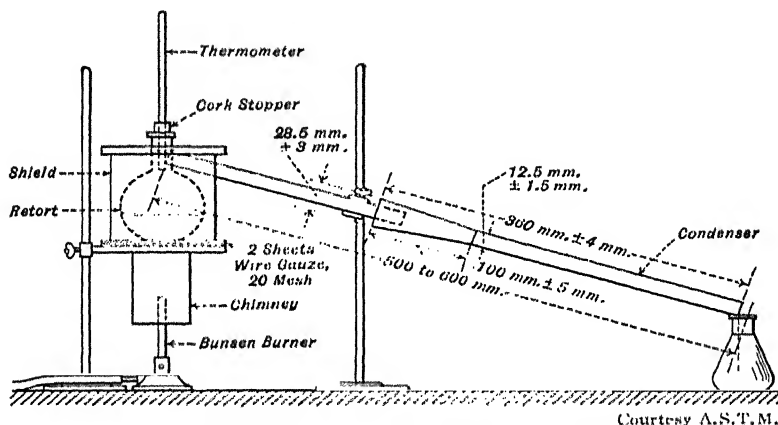


FIG. 311.—Distillation Apparatus Assembly for Road Oils.

in excess of 2.0 per cent, the bituminous material shall be dehydrated before testing, by distilling 500 ml. in an 800-ml. copper still provided with a water-cooled condenser, the distillate being caught in a separatory funnel. When all the water has been expelled, the distillate is allowed to settle, the water drawn off and the oils returned to the residue in the still after the contents have cooled below 212° F.

The flask shall be supported on a tripod or rings over two sheets of 20-mesh gauze, 150 mm. square, as shown in Fig. 311. It shall be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the top of the bulb level with the lowest point of juncture of the tubulature and neck of the flask. The axis of the flask through the neck shall be vertical.

The distance from the bulb of the thermometer to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner should be protected from drafts by a suitable shield or chimney (see Fig. 311).

One hundred grams ($100 \text{ g.} \pm 0.1 \text{ g.}$) of the sample shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from five to fifteen minutes. The distillation shall be conducted at the rate of between 50 and 70 drops per minute and the distillate collected in weighed receivers. (For weighing the receivers and fractions, a balance accurate to at least 0.05 g. shall be used.) The condenser tube shall be warmed whenever necessary to prevent accumulation of solid distillates. The fractions shall be collected at the points designated by the specifications. The receivers shall be changed when the thermometer indicates the maximum temperature for each fraction. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and any oil which has condensed in the condenser tube shall be drained into the last fraction.

The residue shall remain in the flask with the cork and thermometer in position, until no vapors are visible and it shall then be weighed. If tests of the residue are required, the flask shall then be inclined so that the residue will flow around the sides, thus collecting any condensed vapors that may be on the sides of the flask, after which the residue shall be poured into a suitable receptacle and covered. If the residue becomes so cool that it cannot be poured readily from the flask, it shall be reheated to a temperature not exceeding 125°C. by holding the bulb of the flask in a suitable bath and not by the application of flame. During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer. The result of the distillation test shall be reported in percentages by weight of the water-free material. The following fractions are usually reported:

Up to 170°C.
 170 to 235°C.
 235 to 270°C.
 270 to 300°C.
Residue.

Test 16c. For Creosote Oils.¹⁸² In testing creosote, the foregoing method shall be modified as follows: The sample as received should be thoroughly stirred and agitated, warming if necessary to insure a complete mixture free from crystallized solids, before the portion for analysis is removed. The thermometer shall be inserted through a cork in the neck of the flask, with the bottom of its bulb 12 to 13 mm. from the surface of the oil. The exact location of the thermometer bulb shall be determined by placing a vertical rule graduated in divisions not exceeding 1 mm. back of the flask, when the latter is in position for the test, and sighting the level of the liquid and the point for the bottom of the thermometer bulb.

One hundred grams plus or minus 0.1 g. of the sample shall be weighed into the flask, the apparatus assembled, and heat applied so that the first drop falls from the end of the condenser in 10 min. \pm 100 sec. The rate at the end of the condenser shall be adjusted to 90 to 100 drops per min. within 2 min. after the first drop and shall be maintained at 80 to 100 drops per min. throughout the distillation.

The following fractions should be reported: *

0 to 210° C.
210 to 235° C.
235 to 270° C.
270 to 315° C.
315 to 355° C.
Residue.

If the fraction 0 to 210° C. contains water, the amount of water shall be determined. This amount shall then be deducted from the weight of oil taken and all of the fractions shall be corrected to a percentage based on dry oil. A convenient method for determining the amount of water is to transfer this fraction, after weighing, to a tube or cylinder graduated in 0.1 ml. and add about 15 to 20 ml. of benzol. This almost always causes a clear separation between the oil and water.

If the elevation at which the distillation is to be made exceeds 1,000 ft., the temperatures at which the fractions are taken shall be corrected in accordance with Table CXXXV.

*The fraction 235 to 270° C. is not usually required in creosote specifications, but it is recommended that this cut be made, since it gives useful information and does not materially increase the operator's labor.

TABLE CXXXV

TEMPERATURES AT WHICH CREOSOTE FRACTIONS SHOULD BE CUT TO CORRECT DISTILLATION
TEMPERATURES FOR DIFFERENT ALTITUDES
(Corrections made to the nearest 1° C.)

Elevation above Sea Level, ft.	Fractionation Temperatures for Various Altitudes, deg. Cent.					
0	200	210	235	270	315	355
1000	198	208	233	268	313	353
1500	197	207	232	267	312	352
2000	196	206	231	266	311	351
2500	196	206	230	265	310	350
3000	195	205	230	264	309	349
3500	194	204	229	263	308	348
4000	193	203	228	263	307	347
4500	193	202	227	262	306	346
5000	192	202	226	261	305	344
5500	191	201	225	260	304	343
6000	190	200	225	260	303	343

Test 16d. For Cut-back Asphalts. In testing cut-back asphaltic products, the foregoing method has been modified in the following particulars: ¹⁸³

The condenser shall consist of a 250-mm. standard glass-jacketed condenser (Fig. 312), of which the following dimensions are recommended:

Length of jacket, excluding the necks.....	250 mm. ± 5 mm.
Outside diameter of adapter of condenser tube.....	23 mm. ± 1 mm.
Length of adapter.....	75 mm. ± 5 mm.
Outside diameter of condenser tube proper.....	12.5 mm. ± 0.5 mm.
Over-all length of condenser tube, including adapter...	475 mm. ± 25 mm.

The adapter shall be of the curved design of heavy wall (1 mm.) and reinforced top glass, with an angle of approximately 105 deg., and with a diameter at the large end of approximately 18 mm. The outlet end shall be ground to an angle of 45 deg. ± 5 deg. with the inside vertical.

The receivers shall be graduated cylinders, of uniform diameter, with a pressed or molded base and a lipped top. The over-all height shall be not less than 24.8 cm. (9¾ in.) nor more than 26.0 cm. (10¼ in.). The cylinder shall be graduated in single milliliters to contain 100 ml., and the graduated portion shall be not

less than 17.78 cm. (7 in.) nor more than 20.32 cm. (8 in.) in length. Each fifth graduation shall be distinguished by a longer line, and the graduations shall be numbered from the bottom up at intervals of 10 ml. The graduations shall not be in error by more than 1 ml. at any point on the scale.

The flask shall be supported on a tripod or ring over two sheets of 20-mesh gauze, 150 mm. square as shown in Fig. 312. It shall

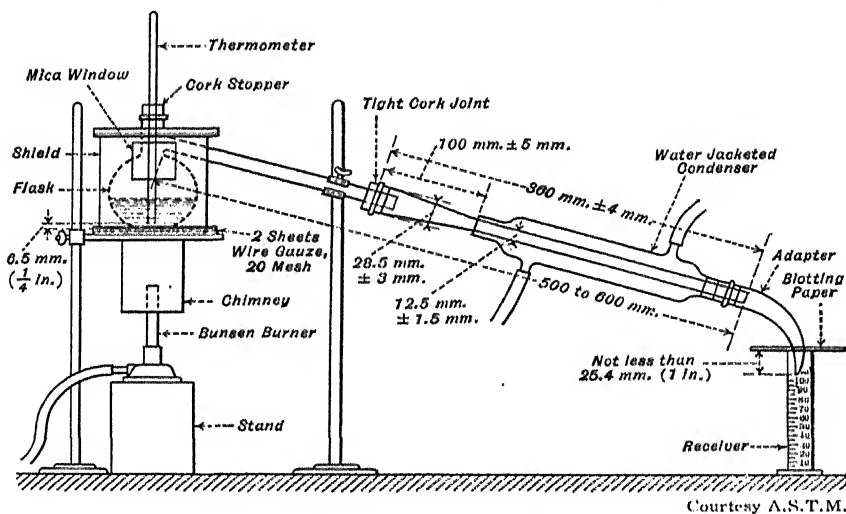


FIG. 312.—Distillation Apparatus Assembly.

be connected to the condenser tube by a tight cork joint. The thermometer shall be inserted through a cork in the neck with the bottom of the bulb $\frac{1}{4}$ in. from the bottom of the flask. The axis of the flask through the neck shall be vertical. The distance from the neck of the flask to the outlet end of the condenser tube shall be not more than 600 nor less than 500 mm. The burner should be protected from drafts by a suitable shield or chimney.

The adapter shall be adjusted over the end of the condenser tube so as to conduct the distillate into the receiver, and the top of the receiver shall be covered closely during the distillation with a piece of blotting paper or its equivalent, which shall be cut so as to

fit the adapter tightly. The adapter shall extend into the receiver at least 2.54 cm. (1 in.) but not below the 100-ml. mark. Unless the laboratory air temperature is between 12.8 and 18.3 C. (55 and 65° F.) the receiver shall be immersed up to the 100-ml. mark in a transparent bath maintained between these temperatures. The condenser tube shall be clean and dry.

Two hundred milliliters of the sample, calculated from the specific gravity of the material at 15.5° C. (60° F.), shall be weighed into the flask, the apparatus assembled and heat applied so that the first drop comes over in from 5 to 15 min. The distillation shall be conducted at the rate of between 50 and 70 drops per minute. Should the sample foam the distillation rate will have to be reduced, but the normal rate shall be resumed as soon as possible. If excess foaming persists the distillation may be more easily controlled by applying the flame near the edge of the bulb instead of at the center of same. The distillate shall be collected in the specified receivers, and the volume of distillate at all specified temperatures recorded. The volume of any separated water shall also be recorded. When the maximum specified temperature of the test is indicated by the thermometer, the flame shall be removed and the residue poured *immediately* into a 6-oz. tin box, 70 mm. (2¾ in.) in diameter and 45 mm. (1¾ in.) in depth, placed on its cover to prevent too rapid cooling at the bottom.

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in dimensions conforming to the above requirements.

Any oil which may remain in the condenser tube shall be drained into the last receiver.

As soon as no further vaporization is apparent, the residue shall be stirred to insure homogeneity, and then poured into the necessary apparatus for the required tests. During the progress of the distillation the thermometer shall remain in its original position. No correction shall be made for the emergent stem of the thermometer. Temperatures to be observed in the distillation test shall be corrected for the effect of the altitude of the laboratory in which the test is made. If the elevation at which the distillation is to be made exceeds 1000 ft., the temperatures at which the

fractions are taken shall be corrected in accordance with Table CXXXV.

The results of the distillation test shall be reported in percentage by volume of water-free material. The following fractions are usually reported:

Up to 225° C. (437° F.),
Up to 315° C. (600° F.),
Up to 360° C. (680° F.).

Additional fractions may be specified, such as:

Up to 160° C. (320° F.),
Up to 175° C. (347° F.),
Up to 190° C. (374° F.).

Standard specifications have also been proposed for testing gas-oil and similar distillate fuel-oils.¹⁸⁴

FLASH-POINT

The flash-point is used primarily for determining the adaptability of bituminous substances for certain definite usages, and serves as a criterion of the fire hazard. It should be at least 50° F. higher than the maximum temperature to which the bituminous substance will be subjected in the process of blending or utilization. This test is also sometimes used for gauging the uniformity of supply and for purposes of factory control. It should be noted, however, that the flash-point of a mixture is always lower than an additive result of computation.

A number of flash-point testers have been proposed,¹⁸⁵ of which the following are most generally used for testing bituminous substances:

Test 17a. Pensky-Martens Tester. This form of tester has been standardized for determining the flash-point of fuel-oil, cut-back asphalts and other viscous materials and suspension of solids,¹⁸⁶ as follows:

The Pensky-Martens tester, a diagram of which appears in Fig. 313, shall include the following major parts:

(a) Cup: The cups shall be made of brass and shall satisfy the following dimensional specifications:

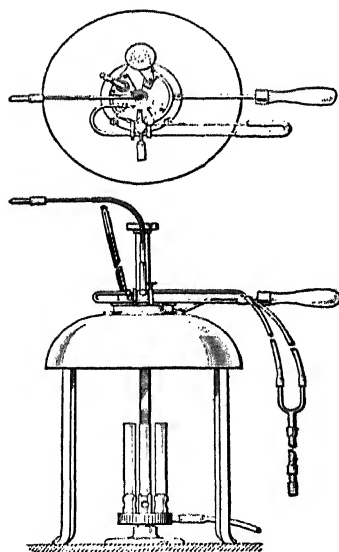
Dimensions	Inches			Centimeters		
	Minimum	Normal	Maximum	Minimum	Normal	Maximum
Inside diameter below filling mark.....	1.950	2.000	2.050	4.953	5.080	5.207
Difference, inside and outside diameters below filling mark..	0.120	0.125	0.130	0.305	0.318	0.330
Inside height.....	2.150	2.200	2.250	5.461	5.588	5.715
Thickness of bottom.....	0.070	0.095	0.120	0.178	0.241	0.305
Distance from rim to filling mark.....	0.845	0.860	0.875	2.146	2.184	2.223
Distance lower surface flange to bottom of cup.....	1.780	1.795	1.810	4.521	4.559	4.597

The inside of the cup may be turned to a slightly larger diameter above the filling mark and the outside may be tapered above the flange, but the wall thickness at the upper edge shall be not less than 0.04 in. (0.102 cm.). The flange should be approximately 0.5 in. (1.27 cm.) wide and approximately 0.125 in. (0.318 cm.)

thick. It shall be equipped with devices for locating the position of the lid on the cup and the cup in the stove. A handle, attached permanently to the flange of the cup, is a desirable accessory.

(b) Lid.

(c) Stirring Device: The lid shall be equipped with a stirring device consisting of a vertical steel shaft, not less than 0.1 in. (0.254 cm.) nor more than 0.125 in. (0.318 cm.) in diameter, mounted in the center of the cup, and carrying two two-bladed brass propellers. The blades of both propellers shall be approximately 0.313 in. (0.795 cm.) wide and shall be set at an angle of approximately 45 deg. The smaller (upper) propeller shall have an over-all diameter of approximately 0.75 in. (1.905 cm.). The



Courtesy A.S.T.M.

FIG. 313.—The Pensky-Martens Tester.

larger (lower) propeller shall have an over-all diameter between 1.25 and 1.75 in. (3.175 and 4.445 cm.). The thickness of the propeller blades shall be not less than 0.057 in. (0.145 cm.) nor more than 0.081 in. (0.206 cm.), which limits correspond respectively to No. 15 and No. 12 B. and S. gage sheet brass. The collars on which the propeller blades are mounted shall have horizontal and vertical dimensions not greater than 0.4 in. (1.016 cm.).

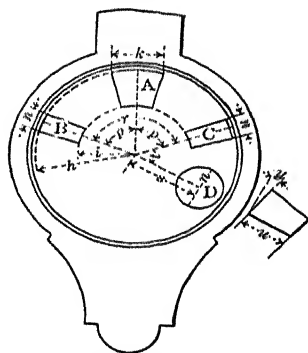
The plane of the center of the upper propeller shall be 0.4 in. (1.016 cm.) below the level of the rim of the cup. The plane of the center of the lower propeller shall be 2.0 in. (5.08 cm.) below the level of the rim of the cup. The level of the rim of the cup is in effect the level of the plane part of the portion of the lower surface of the lid inside the rim.

(d) Cover Proper: The cover proper shall be of brass and shall have a rim projecting downward almost to the flange of the cup and fitting the outside of the cup closely. The thickness of the cover, measured just inside the rim, shall be not less than 0.031 in. (0.079 cm.) nor more than 0.078 in. (0.198 cm.). There shall be a proper locating device engaging with a corresponding locating device on the flange of the cup.

There shall be four openings in the cover, as indicated in Fig. 314.

Opening *A* is an area defined by arcs of two concentric circles and the intersected lengths of two radii. The radius of the outer circle shall be not less than 0.938 in. (2.383 cm.) nor more than 0.969 in. (2.461 cm.). The radius of the inner circle shall not be less than 0.531 in. (1.349 cm.) nor more than 0.563 in. (1.430 cm.). The chord of the arc of the outer circle shall not be less than 0.500 in. (1.270 cm.) nor more than 0.540 in. (1.372 cm.).

Openings *B* and *C* are equal areas, each of the same general



<i>h</i>	Minimum 0.938 in., Maximum 0.969 in.
<i>r</i>	" 0.531 " " 0.563 "
<i>k</i>	" 0.500 " " 0.540 "
<i>l</i>	" 0.167 " " 0.219 "
<i>g</i>	Approximately 0.75 in.
<i>u</i>	0.5 in.
Angles <i>p</i>	Equal
Angle <i>r</i>	Min. 135°; Max. 140°
Angle <i>t</i>	" 50°; " 60°
Angle <i>u</i>	" 10°; " 15°

FIG. 314.—Cover for Pensky-Martens Tester.

form as opening *A* but of approximately half the (angular) width. The radii of the defining inner and outer circles shall be within the limits specified for the radii of the two circles, arcs of which partly define opening *A*. The chord of the outer arc for opening *B* or opening *C* shall not be less than 0.187 in. (0.475 cm.) nor more than 0.219 in. (0.556 cm.). The sum of the areas of openings *B* and *C* shall not be less than 75 per cent nor more than 100 per cent of the area of opening *A*. Openings *B* and *C* shall be equally distant from opening *A* and radii drawn through each of their centers shall be at an angle of not less than 135 deg. nor more than 140 deg.

Openings *A*, *B*, and *C* need not conform exactly to the shape of geometrical figures bounded by arcs of two concentric circles and intersected lengths of radii. Their boundaries must, however, fall on or between the lines indicated by the limiting values of the dimensional specification of the preceding text and of Fig. 314.

Opening *D* is for a thermometer collar. Its center is approximately 0.75 in. (1.905 cm.) from the center of the lid and on a radius at an angle of not less than 50 deg. nor more than 60 deg. from a radius passing through the center of opening *C*. The thermometer collar shall have an inside diameter of approximately 0.5 in. (1.27 cm.). It shall be set at an angle of not less than 10 deg. nor more than 15 deg. from the perpendicular.

(*e*) Shutter: The lid shall be equipped with a brass shutter approximately 0.094 in. (0.239 cm.) thick, operating on the plane of the upper surface of the lid. The shutter shall be so shaped and mounted that it rotates on the axis of the horizontal center of the lid between two stops, so placed that when in one extreme position the openings *A*, *B*, and *C* of the lid are completely closed and when in the other extreme position these orifices are completely opened.

(*f*) Flame Exposure Device: The flame exposure device shall have a tip with an opening 0.027 in. (0.069 cm.) to 0.031 in. (0.079 cm.) in diameter. The flame exposure device shall be equipped with an operating mechanism which, when the shutter is in the "open" position, depresses the tip so that the center of the orifice is between the planes of the under and upper surfaces of the lid proper, at a point on a radius passing through the center of the larger opening *A* and approximately 0.1 in. (0.254 cm.) from the outer edge of the opening.

NOTE.—A pilot flame for automatic relighting of the exposure flame should be provided.

A bead 0.156 in. (0.396 cm.) in diameter, of some suitable material, may be mounted on the lid so that the size of the test flame can be regulated by comparison.

The mechanism operating the shutter should be of the spring type and constructed so that when at rest the shutter shall exactly close the three openings. When operated to the other extreme, the three openings in the lid shall be exactly open and the tip of the exposure tube shall be fully depressed.

(g) Stove: Heat shall be supplied to the cup by means of a properly designed stove which is equivalent to an air bath. This stove shall consist of (1) an air bath and (2) a top plate on which the flange of the cup rests.

(h) Air Bath: The air bath shall have a cylindrical interior 1.625 in. (4.128 cm.) to 1.656 in. (4.206 cm.) deep and a diameter not less than 0.125 in. (0.317 cm.) nor more than 0.156 in. (0.396 cm.) greater than the outside diameter of the cup. The air bath may be either a flame-heated metal casting or an electric resistance element.

NOTE.—If the heating element is a flame-heated metal casting, it shall be so designed and used that the temperature of bottom and walls is approximately the same. On this account it should be not less than 0.25 in. (0.635 cm.) thick. The casting shall be designed so that products of combustion of the flame cannot pass up and in contact with the cup.

If the air bath is of the electric-resistance type, it shall be constructed so that all parts of the interior surface are heated equally. This necessitates an even distribution of resistance wire over bottom and walls and a method of construction such that heat is given out from the whole core of the resistance element rather than directly from the wire.

(i) Top Plate: The top plate shall be of metal. The total distance from the upper surface of the plate to the bottom of the air bath shall exceed the distance from the under surface of the flange to the bottom of the cup by not less than 0.063 in. (0.160 cm.) nor more than 0.125 in. (0.317 cm.). The top plate shall be mounted with an air gap between it and the air bath. The top plate may be attached to the air bath by means of three screws and spacing-bushings. The spacing-bushings should be of proper thickness to define the air gap which shall be not less than 0.125 in. (0.317 cm.) nor more than 0.187 in. (0.475 cm.). The spacing-bushings shall be not more than 0.375 in. (0.952 cm.) in diameter.

(j) Thermometers: Two standard thermometers shall be used with the A.S.T.M. Pensky-Martens tester. The low range, "P.M. and Tag" thermometer shall be used for tests when the indicated

reading falls within the limits 20 to 200° F. The "P.M. high" thermometer shall be used for tests when the indicated reading falls within the limits 230 to 700° F. For the range 200 to 230° F. either thermometer may be employed, depending on the convenience of the operator. The low-range "P.M. and Tag" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, of ranges -7 to +110° C. or +20 to +230° F., respectively. The high-range "P.M. High" thermometer specifications cover a special thermometer graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being 90 to 370° C. or 200 to 700° F., respectively. Thermometers shall be mounted so that the bottom of the bulb is 1.75 in. (4.445 cm.) below the level of the rim of the cup (which corresponds to the level of the lower surface of the portion of the lid inside the rim).

All parts of the cup and its accessories shall be thoroughly clean and dry before the test is started. Particular care shall be taken to avoid the presence of any gasoline or naphtha used to clean the apparatus after a previous test. The cup shall be filled with the oil to be tested up to the level indicated by the filling mark.

The lid shall be placed on the cup and the latter set in the stove. Care shall be taken to have the locating devices properly engaged. The thermometer shall be inserted. If it is known that the oil will flash above 220° F., the "P.M. High" thermometer may be selected; otherwise, it is preferable to start with the "P.M. and Tag" thermometer and change in case a temperature of 220 to 230° F. is reached.

The test flame shall be lighted and adjusted so that it is of the size of a bead $\frac{5}{32}$ in. (3.97 mm.) in diameter. Heat shall be supplied at such a rate that the temperature read on the thermometer increases not less than 9 nor more than 11° F. per minute. The stirrer shall be turned at a rate of from 1 to 2 revolutions per second. Application of the test flame shall be made at each temperature reading which is a multiple of 2° F. up to 220° F. For the temperature range above 220° F., application shall be made at each temperature reading which is a multiple of 5° F. The first application of the test flame shall be made at a temperature at least 30° F. below the actual flash-point. Application of the test

flame shall be made by operating the device controlling the shutter and test-flame burner so that the flame is lowered in one-half second, left in its lowered position for one second, and quickly raised to its high position. Stirring shall be discontinued during the application of the test flame.

The flash-point is taken as the temperature read on the thermometer at the time of the flame application that causes a distinct flash in the interior of the cup. The true flash must not be confused with the bluish halo that sometimes surrounds the test flame for the applications preceding the one that causes the actual flash.

The barometric pressure shall be observed and recorded. No corrections shall be made except in case of dispute, when the flash-point figure shall be corrected according to the following rule: For each inch (25 mm.) below 29.92 in. (760 mm.) barometric reading, add 1.6° F. to the flash-point. For each inch (25 mm.) above 29.92 in. (760 mm.) barometric reading, subtract 1.6° F. from the flash-point.

Determination of Flash-point of Cut-back Asphalts and Other Viscous Materials and Suspensions of Solids. The apparatus shall consist of the following:

(a) Pensky-Martens tester as described above, except that the stirrer shall be mechanically operated to stir in a downward direction at a speed of 70 to 80 r.p.m.

(b) Low-range thermometer as specified.

The material to be tested and the tester shall be brought to a temperature 30° F. lower than the estimated flash-point. The air space between the cup and the interior of the air bath shall be completely filled with water at the temperature of the tester and material. The temperature shall be raised throughout the duration of the test at a rate of not less than 2 nor more than 3° F. per min. With the exception of this requirement for rate of heating and the rate of stirring of 70 to 80 r.p.m., the procedure shall be the same as that prescribed in the Standard Method of Test for Flash-point by Means of the Tag Closed Tester (Test 17c).

Duplicate tests should not differ by more than 5° F.

A simplified form of Pensky-Martens tester for approximately determining the flash-point, consists of a glass beaker or metal cup having the same dimensions, namely 5.0 cm. in diameter, and 5.5

cm. in depth, filled to within 2.1 cm. of its upper rim with the material to be tested. This is supported on a sand bath and the thermometer bulb immersed in the bituminous material without, however, touching the sides or bottom. The test flame is adjusted to a 3 mm. cross-section, and the test performed exactly is described for the Pensky-Martens tester.¹⁸⁷

Test 17b. Cleveland Tester. This tester has also been standardized as follows:¹⁸⁸

The open-cup flash and fire test on all products except fuel oils and those having an open cup flash below 175° F. (79° C.) shall be determined in the Cleveland Open Cup.

(a) The cup illustrated in Fig. 315 shall be made of brass and shall conform to the requirements in Table CXXXVI.

TABLE CXXXVI
DIMENSIONS OF CLEVELAND OPEN CUP

	Inches			Centimeters		
	Min.	Normal	Max.	Min.	Normal	Max.
Inside diameter immediately below filling mark.....	2 $\frac{11}{32}$	2 $\frac{1}{2}$	2 $\frac{11}{32}$	6.27	6.35	6.43
Outside diameter below flange.....	2 $\frac{11}{32}$	2 $\frac{11}{32}$	2 $\frac{11}{32}$	6.75	6.83	6.91
Inside height from center of bottom to rim.....	1 $\frac{9}{32}$	1 $\frac{5}{8}$	1 $\frac{11}{32}$	3.25	3.33	3.41
Thickness of bottom.....	$\frac{3}{4}$	$\frac{1}{2}$	$\frac{3}{4}$	0.28	0.32	0.36
Distance from rim to filling mark.....	$\frac{11}{32}$	$\frac{1}{2}$	$\frac{11}{32}$	0.91	0.95	0.99
Distance lower surface flange to bottom of cup.....	1 $\frac{1}{2}$	1 $\frac{1}{2}$	1 $\frac{1}{2}$	3.10	3.18	3.26
Vertical distance upper surface flange to rim.....	$\frac{3}{4}$	$\frac{1}{2}$	$\frac{3}{4}$	0.28	0.32	0.36
Thickness of rim.....	$\frac{3}{4}$	$\frac{3}{8}$	$\frac{3}{4}$	0.20	0.24	0.28
Width of lower surface of flange.....	$\frac{9}{16}$	$\frac{11}{32}$	$\frac{1}{2}$	1.43	1.51	1.59

The bevelled edge of the cup shall be at an angle of approximately 45 deg. There may be a fillet of approximately $\frac{5}{32}$ in. (0.397 cm.) radius inside the bottom of the cup.

(b) Heating Plate: The cup shall be supported by a metal plate (Fig. 316) $\frac{1}{4}$ in. (0.635 cm.) in thickness and 6 in. (15.24 cm.) in width. The plate shall be of brass, cast iron, wrought iron or steel. In the center of the plate there shall be a plane depression $\frac{1}{32}$ in. (0.079 cm.) in depth, and of just sufficient diameter to fit

the cup. There shall be a circular opening $2\frac{3}{16}$ in. (5.50 cm.) in diameter, cut through the plate, centering with the center of the above-mentioned depression. The plate shall be covered with a sheet of hard asbestos board $\frac{1}{4}$ in. in thickness, and of the same shape as the metal plate. There shall be cut in the center of the asbestos board a circular hole just fitting the cup. Heat may be supplied from any convenient source. The use of a gas burner,

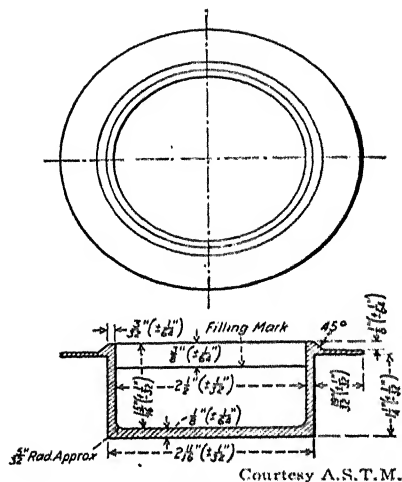


FIG. 315.—Cleveland Open Cup.

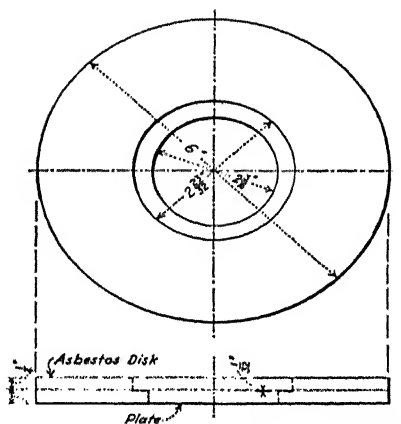


FIG. 316.—Heating Plate.

electric heater, or alcohol lamp is permitted, but under no circumstances are products of combustion or free flame allowed to come up around the cup. The source of heat shall be centered under the opening in the plate and shall be of a type that will not produce local superheating. If a flame heater is used, it may be protected from drafts or excessive radiation by any suitable type of shield, that does not project above the level of the upper surface of the asbestos board.

(c) The thermometer shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being -6 to $+400^{\circ}$ C., or $+20$ to $+760^{\circ}$ F., respectively.

The thermometer shall be suspended or held in a vertical position by any suitable device. The bottom of the bulb shall be $\frac{1}{4}$ in.

(0.635 cm.) from the bottom of the cup, and above a point half way between the center and back of the cup.

The cup shall be filled with the oil to be tested in such a manner that the top of the meniscus is exactly at the filling line at room temperature. When asphalt or other solid bituminous material is to be tested, it shall first be heated to a temperature not less than 300° F. nor more than 350° F., to render it sufficiently fluid. The cup shall then be filled with the hot fluid at this temperature in the same manner as with oil. The subsequent procedure shall then be the same for both asphalt and solid bituminous material as with oil.

The surface of the oil shall be free from bubbles. There shall be no oil above the filling line or on the outside of the apparatus. The test flame shall be approximately $\frac{5}{32}$ in. (0.397 cm.) in diameter.

NOTE.—For purposes of comparison it is recommended that a bead of suitable light-colored material be mounted in a convenient position so that the size of the test flame can be determined. The device for applying the flame may be of any suitable type, but it is suggested that the tip be approximately $\frac{1}{16}$ in. (0.159 cm.) in diameter at the end and that the orifice be $\frac{1}{32}$ in. (0.079 cm.) in diameter. If the device for operating the test flame be mounted in such a manner as to permit automatic duplication of the sweep of the test flame, the radius of swing shall be not less than 6 in.

The test flame shall be applied as the temperature read on the thermometer reaches each successive 5° F. mark. The flame shall pass in a straight line (or on the circumference of a circle having a radius of at least 6 in.) across the center of the cup and at right angles to the diameter passing through the thermometer. The test flame shall, while passing across the surface of the oil, be in the plane of the upper edge of the cup. The time for the passage of the test flame across the cup shall be approximately one second.

The oil shall be heated at a rate not exceeding 30° F. per minute temperature rise, till a point is reached approximately 100° F. below the probable flash-point of the oil. Thereafter the rate of heating shall be decreased and for at least the last 50° F. before the flash-point is reached, the rate shall be not less than 9 nor more than 11° F. per minute.

The flash-point shall be taken as the temperature read on the thermometer when a flash appears at any point on the surface of

the oil. The true flash must not be confused with a bluish halo that sometimes surrounds the test flame.

After determining the flash-point, the heating shall be continued at the specified rate of 9 to 11° K. per minute, and application of the test flame shall be made at the specified intervals until the oil ignites and continues to burn for a period of at least five seconds. The method of application of the flame shall be the same as for flash-point. The temperature read at the time of the flame application which causes burning for a period of five seconds or more shall be recorded as the fire-point. The flash-point and fire-point tests shall be made in a room or compartment free from air drafts. The operator shall avoid breathing over the surface of the oil. It is desirable that the room or compartment be darkened sufficiently so that the flash may be readily discernible.

Test 17c. Tag Closed Tester. This test has also been standardized¹⁸⁰ as follows:

The Tag Closed Tester shall be used for all volatile flammable liquids flashing below 175° K. with the exception of products classed as fuel oil, which are preferably tested with the Pensky-Martens Closed Tester. Provision is made in this method for determining the flash-point of lacquer solvents or diluents of the low flash-points.

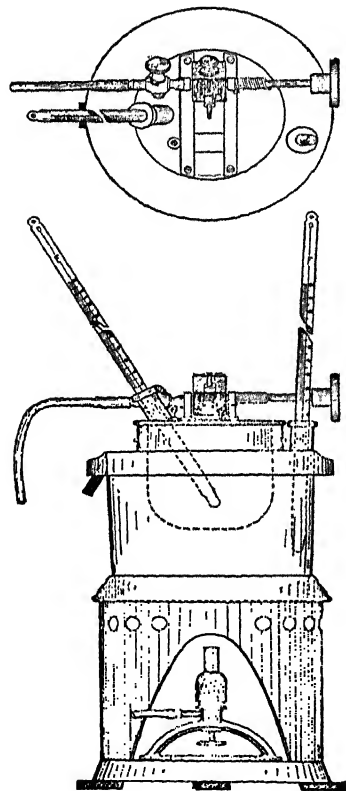
(a) The Tag Closed Tester, a diagram of which is shown in Fig. 317, shall conform to the dimensions within the limits of tolerances given in Table CXXXVII.

TABLE CXXXVII
DIMENSIONS OF TAG CLOSED CUP

Dimension	Normal	Tolerance
Depth of water surface below top of cup, in. .	1 $\frac{3}{8}$ (27.8 mm.)	$\pm \frac{1}{8}$ (0.4 mm.)
Depth of oil surface below top of cup, in. . . .	1 $\frac{3}{8}$ (29.4 mm.)	$\pm \frac{1}{8}$ (0.4 mm.)
Depth of top of bulb of oil thermometer when in place below top of cup, in.	1 $\frac{5}{8}$ (33.3 mm.)	$\pm \frac{1}{8}$ (0.8 mm.)
Inside diameter of oil cup at top, in.	2 $\frac{1}{2}$ (54.0 mm.)	± 0.005 (0.1 mm.)
Diameter of bead on top of cover, in.	$\frac{5}{8}$ (4.0 mm.)	$\pm \frac{1}{8}$ (0.4 mm.)
Weight of oil cup, g.	68	± 1

The plane of underside of cover to be between the top and bottom of the burner tip when the latter is fully depressed.

(b) Two thermometers are required, one for measuring the temperature of the sample under test, the other for measuring the temperature of the bath. Each of these thermometers shall conform to the following requirements, namely they shall be graduated in either Centigrade or Fahrenheit degrees as specified, the ranges being -7 to $+110^{\circ}$ C. or $+20$ to 230° F., respectively.



Courtesy A.S.T.M.

FIG. 317.—Tag Closed Tester.
(Arranged for Use of Gas.)

The test shall be performed in a room or compartment darkened sufficiently to permit ready detection of the flash. Care shall be taken to have the tester level and steady. It shall be surrounded on three sides by an enclosure for protection from drafts. (A shield 18 in. square and 24 in. in height, open in front, is suggested. Tests made in a laboratory hood or near ventilators are not to be relied upon.)

Gas may be used for the test flame and for heating the water bath. If gas is not available for the test flame, a wick of cotton cord may be inserted in the burner tip, a small quantity of cotton waste placed in the oil chamber to which the burner tip is attached and the chamber filled with signal, sperm, or lard oil. An alcohol lamp may be used for heating the water bath as a substitute for gas.

The water-bath thermometer shall be placed in the collar provided for it and the bath filled with water until it overflows. The temperature of the water in the bath shall be such that when testing is started it will be at least 20° F. (11° C.) below the probable flash-point of the oil to be tested.

The oil cup shall be placed in its proper position in the water

bath and 50 ml. of the oil to be tested shall be measured into it, using an accurate graduate or other measuring device for the purpose. The temperature of the oil shall be at least 20° F. (11° C.) below its probable flash-point when the test is started. Air bubbles on the surface of the oil shall be destroyed, and the cover with the flash-point thermometer in place shall then be properly attached to the bath collar. The test flame shall be lighted, the flame being adjusted to the size of the small white bead on the cover.

The gas burner or alcohol lamp shall be centrally placed in the base of the tester and lighted. The flame shall be so adjusted that the temperature of the oil in the cup rises at the rate of 1.8° F. (1° C.) per min. as closely as possible, but in any case not faster than 2° F. (1.1° C.) nor slower than 1.6° F. (0.9° C.) per min.

The barometric pressure shall be recorded. If a barometer is not available, the figure may be obtained from the nearest Weather Bureau Station and an appropriate correction made for difference in altitude between such station and the laboratory. The initial temperature of the oil shall be recorded. When the temperature of the oil is 9° F. (5° C.) below its probable flash-point, the knob on the cover shall be turned in such a manner as to introduce the test flame into the vapor space of the cup, and *immediately* turned back again. The time consumed in turning the knob down and back shall be about one full second, or the time required to pronounce distinctly the words "thousand and one." The time at which the first introduction of the test flame is made and the temperature of the oil shall be recorded.

The application of the test flame shall be repeated after each 1° F. (0.5° C.) rise in temperature of the oil until a distinct flash in the interior of the cup is observed. The true flash must not be confused with the bluish halo which sometimes surrounds the test flame during applications immediately preceding the actual flash.

The time and the temperature of the oil when the flash-point is reached shall be recorded.

If the rise in temperature of the oil from the time of making the first introduction of the test flame to the time at which the flash-point is observed was more rapid than 2° F. (1.1° C.), or slower than 1.6° F. (0.9° C.) per min., the test shall be repeated, adjusting the gas burner or alcohol lamp to the proper rate of heating.

It is not necessary to turn off the test flame with the small regulating valve on the cover; it may be left adjusted to the proper size of flame.

After completing the preliminary test to determine the approximate flash-point, the burner or lamp shall be removed, the oil cover lifted and the thermometer bulb carefully wiped off. The oil cup shall be removed, emptied, and carefully wiped until dry. The temperature of the bath shall be lowered by the addition of cold water until it is 15°F. (8°C.) below the flash-point of the oil as shown by the preliminary test. The oil cup shall be replaced and a fresh 50-ml. sample measured into it. The test procedure, as described above, shall then be repeated, introducing the test flame for the first time, however, when the oil temperature is 10°F. (5.5°C.) below the flash-point obtained in the preliminary test.

Oil which has once been subjected to the flash test shall be discarded. If test is to be repeated, a fresh sample shall be used. If two or more determinations agree within 1°F. (0.5°C.), the average of these results, corrected for barometric pressure, shall be considered the flash-point. If two determinations do not check within 1°F. (0.5°C.), a third determination shall be made and, if the maximum variation of the three tests is not greater than 2°F. (1°C.), their average, after correcting for barometric pressure, shall be considered the flash-point.

Correction for barometric pressure shall be made only in cases of dispute or when the barometer reading varies more than $\frac{1}{2}$ in. (13 mm.) from the standard pressure of 29.92 in. (760 mm.). When the barometer reading is below this standard pressure, add to the thermometer reading 1.6°F. (0.9°C.) for each inch (25 mm.) of barometer difference to obtain the true flash-point. When the barometer reading is above the standard pressure, deduct 1.6°F. (0.9°C.) for each inch (25 mm.) of barometer difference to obtain the true flash-point.

Determination of Flash-point of Lacquer Solvents or Diluents of Low Flash-point. The apparatus shall consist of the following:

- (a) Flash-point Tester, as described above.
- (b) Thermometer: For determining the flash-points below 40°F. (4.4°C.), suitable thermometers of low range shall be used. These thermometers shall conform to the following ranges, 0 to

120° F. or -20° C. to 50° C. These thermometers may not be used in the testing of materials flashing above 70° F. (21.1° C.).

The procedure to be followed shall be the same as that described above, except that in determining flash-points below 40° F. (4.4° C.) the bath shall be filled with brine or other low-freezing liquid instead of water.

Test 17d. Tag Open Tester. The Tagliabue tester may be used as an open tester (i.e., without the cover) for examining bituminous road oils, in which case the test is performed as described in Test 17c, the cover being omitted.

The following specifications have been formulated for use with materials having a flash-point of less than 175° F.:¹⁰⁰

(a) The Tagliabue open-cup flash tester shall be used. The instrument shall set firm and level.

(b) Fill the metal bath cup with water having a temperature at least 20° F. below the probable flash-point of the material to be tested, leaving room for displacement by the glass oil cup, which is then placed in the bath.

(c) Fill the glass oil cup with the material to be tested to within $\frac{5}{16}$ in. of its upper level edge. See that there is none of the bituminous material on the outside of the cup, or upon its upper level edge, using soft paper to clean the cup.

(d) Adjust the horizontal flashing-taper guide wire in place. Suspend the thermometer, with its bulb well covered by the material. Heat bath with small flame lamp, having the flame so adjusted that it will raise the temperature of the material at a rate of 2½° F. per minute, without removing the lamp during the whole operation.

(e) For viscous liquids it is necessary that the liquid be stirred at intervals during the test.

(f) Remove air bubbles, if any, from the surface of the material before first trial for flash is made.

(g) At the proper trial temperatures, noted below, try for flash with a small bead of flame (not over $\frac{1}{8}$ in.) by drawing it quickly and without pause across the guide wire from left to right.

(h) The temperature at which the first or initial flash is obtained is called the "flash-point."

(i) Trial temperatures: For materials which may be expected to flash at about 80° F., try for flash at 70° F., then at 75°, 77°, 81°, 83°, and 85°. For other materials try for flash first at a temperature about 20° F. below the expected flash-point and then try for flash at every 5° F.

BURNING-POINT

Test 18. Conventional Method. The burning-point is used to supplement the flash-point, and is of value in determining the adaptability of bituminous substances for particular purposes, from the standpoint of fire hazard. The test may be performed in any of the apparatus described under flash-point (Test 17). In determining the burning-point, the cover of the tester is removed, and the heating, also exposure to the test flame continued in the same manner as for the flash-point, until the vapors ignite and continue to burn.

FIXED CARBON

This test is used solely for purposes of identification, and is especially useful in differentiating the asphaltites, the asphaltic pyrobitumens, and the non-asphaltic pyrobitumens.

The test is a modification of the standard method for ascertaining the fixed carbon of coal.¹⁰¹ There are, however, two schools of thought as to the procedure. One favors a slow initial heating of the substance to expel most of the volatile constituents without causing foaming, before applying the coal method. The other recommends the use of a crucible with a 2-mm. hole in the cover to permit the free escape of the volatile constituents and applying the full heat immediately.¹⁰² It has also been suggested that the sample (1 gram) be covered with sand (10 grams) before ignition to inhibit entrainment of volatile matter.¹⁰³ The author favors the former procedure, patterned along the method of ascertaining the coke residue of creosote oil.

In the presence of mineral matter, the percentage of fixed carbon should be calculated on the basis of the non-mineral constituents. Mineral matter does not vitiate the results but merely acts as a diluent. Thus a pure grahamite containing 0.2 per cent mineral matter and 52.22 per cent fixed carbon, when mixed with an equal weight of clay, tested 26.33 per cent, equivalent to 52.7 per cent fixed carbon on the basis of the non-mineral constituents present.

Test 19. Carbon Residue of Creosote. The following method has been standardized¹⁰⁴ for ascertaining the fixed carbon (coke residue) in creosote or creosote coal-tar solution by coking the resi-

due resulting from the distillation test (Test 20) and calculating the result based on the original oil.

(a) Crucible: A platinum crucible shall be used, with tightly fitting cover of the inverted or capsule type having a depth of about 1 cm., provided with a hole 2 mm. in diameter at its center. The crucible shall have a capacity of 25 to 30 ml. and with cover shall weigh 25 to 30 g.

(b) Furnace: A vertical electric tube furnace of the form shown in Fig. 318 or a Bunsen or Meker burner may be used.

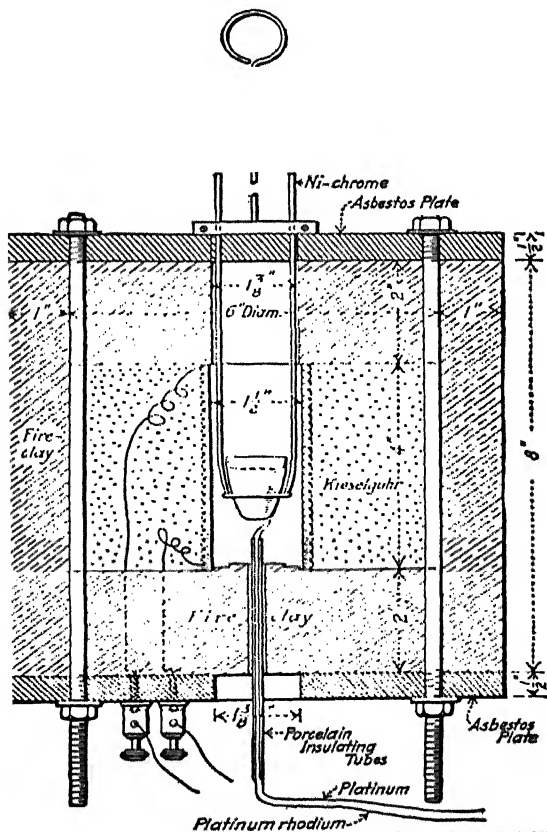


FIG. 318.—Electric Furnace for Determining Coke Residue.

The residue resulting from the distillation test shall be poured directly into the tared crucible or into a tin box wherein it may be heated on a water or steam bath, but not over a flame. One gram ± 0.1 g. of the residue shall be weighed into the covered crucible.

If the vertical electric tube furnace is used, the temperature of the furnace shall be controlled by a thermocouple at $950^{\circ}\text{C.} \pm 20^{\circ}\text{C.}$ The crucible shall be suspended in the electric furnace for exactly 7 minutes, after which it shall be removed, cooled and weighed.

If a Bunsen or Meker burner is used, it shall have a free flame 20 cm. in height. The crucible shall set in a Nichrome triangle with approximately two-thirds of its height below the triangle and with the bottom of the crucible 6 to 8 cm. above the top of the burner. Assurance of the desired temperature of $950^{\circ}\text{C.} \pm 20^{\circ}\text{C.}$ may be indicated by the fusion of crystals of potassium chromate in the crucible when exposed in the specified position for the test. The crucible and contents shall be exposed to the full flame of the burner for exactly 7 minutes. The test shall be conducted in a part of the laboratory free from draughts.

The percentage of coke obtained from the residue in accordance with this test shall be calculated to percentage of total substance by the following formula:

$$\text{Coke in oil} = \frac{A \times B}{100}$$

where A = the percentage residue from oil distilled to 355°C. ,
 B = the percentage of coke in the residue.

Test 20. Carbon Residue of Petroleum Products. Several methods have been standardized for ascertaining the carbon residue of petroleum products, including:

(1) *Conradson Test for Carbon Residue*, which is conducted as follows:¹⁹⁸

This method of test is a means of determining the amount of carbon residue left on evaporating an oil under specified conditions, and is intended to throw some light on the relative carbon-forming propensity of an oil. The results of the test must be considered in connection with other tests and the use for which the oil is intended.

This test furnishes pertinent information relative to lubricants for internal combustion engines, domestic oil fuels, and oils used in the manufacture of gas.

The apparatus (see Fig. 319) shall consist of the following:

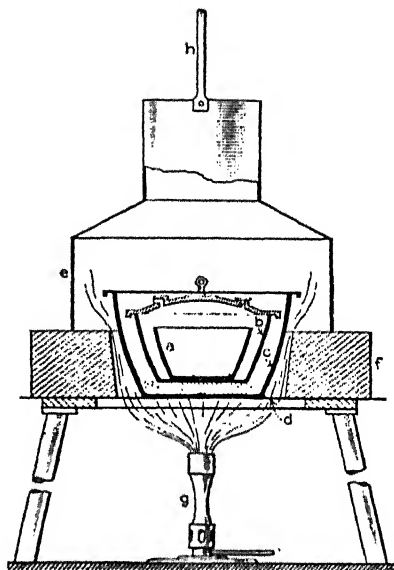
(a) Porcelain Crucible: Porcelain crucible, *a*, wide form, glazed throughout, or a silica crucible; 29- to 31-ml. capacity, 46 to 49 mm. (1.81 to 1.93 in.) in rim diameter.

(b) Iron Crucible: Skidmore iron crucible, *b*, flanged and ringed, 65- to 82-ml. capacity, 53 to 57 mm. (2.07 to 2.20 in.) inside and 60 to 67 mm. (2.36 to 2.64 in.) outside diameter of flange, 37 to 39 mm. (1.46 to 1.54 in.) in height supplied with a cover without delivery tubes and having the vertical opening closed. The horizontal opening of about 6.5 mm. (0.26 in.) shall be kept clean. The outside diameter of the flat bottom shall be 30 to 32 mm. (1.18 to 1.26 in.).

(c) Iron Crucible: Spun sheet-iron crucible, *c*, with cover; 78 to 82 mm. (3.07 to 3.23 in.) in outside diameter at the top, 58 to 60 mm. (approximately 2.3 in.) in height, and approximately 0.8 mm. (0.03 in.) in thickness. Place at the bottom of this crucible, and level before each test, a layer of about 25 ml. of dry sand, or enough to bring the Skidmore crucible, with cover on, nearly to the top of the sheet-iron crucible.

(d) Wire Support: Triangle of bare Nichrome wire, *d*, of approximately No. 13 B. & S. gage having an opening small enough to support the bottom of the sheet-iron crucible at the same level as the bottom of the asbestos block or hollow sheet-metal box, paragraph (f).

(e) Hood: Circular sheet-iron hood, *e*, from 120 to 130 mm. (4¾ to 5¼ in.) in diameter the height of the lower perpendicular side to be from 50 to 53 mm. (2 to 2½ in.); provided at the top



Courtesy A.S.T.M.

FIG. 319.—Apparatus for Determining Carbon Residue.

with a chimney 50 to 60 mm. (2 to 2½ in.) in height and from 50 to 56 mm. (2 to 2¼ in.) in inside diameter, which is attached to the lower part having the perpendicular sides by a cone-shaped member, bringing the total height of the complete hood from 125 to 130 mm. (4⅞ to 5⅛ in.). The hood may be made from a single piece of metal provided the foregoing dimensions are adhered to. As a guide for the height of the flame above the chimney, a bridge made of approximately 3-mm. (¼ in.) iron or Nichrome wire, *h*, shall be attached having a height of 50 mm. (2 in.) above the top of the chimney.

(*f*) Insulator: Asbestos block, refractory ring, or hollow sheet-metal box, 150 to 175 mm. (6 to 7 in.) in diameter if round or on a side if square, 32 to 38 mm. (1¼ to 1½ in.) in thickness, provided with a metal-lined, inverted cone-shaped opening through the center; 83 mm. (3¼ in.) in diameter at the bottom, and 89 mm. (3½ in.) in diameter at the top. In the case of the refractory ring no metal lining is necessary, providing the ring is of hard, heat-resistant material.

(*g*) Burner: Burner, *g*, Meker type, 24 mm. (1 in.) in diameter by 155 mm. (6 in.) in height, suitable for either manufactured or natural gas.

Weigh to the nearest 5 mg. a 10-g. sample of the oil to be tested, free of moisture and other suspended matter, into a tared porcelain or silica crucible containing two glass beads about 0.1 in. in diameter. Place this crucible in the center of the Skidmore crucible. Level the sand in the large sheet-iron crucible and set the Skidmore crucible on it in the exact center of the iron crucible. Apply covers to both the Skidmore and the iron crucible, the one to the latter fitting loosely to allow free exit to the vapors as formed.

On a suitable stand or ring, place the bare Nichrome wire triangle and on it the insulator. Next, center the sheet-iron crucible in the insulator with its bottom resting on top of the triangle, and cover the whole with the sheet-iron hood in order to distribute the heat uniformly during the process.

Apply heat with a high, strong flame from the Meker-type gas burner, so that the pre-ignition period will be 10 ± 1.5 min. (a shorter time may start the distillation so rapidly as to cause foaming or too high a flame). When smoke appears above the chimney, immediately move or tilt the burner so that the gas flame plays on

the sides of the crucible for the purpose of igniting the vapors. Then remove the heat temporarily, and before replacing adjust by screwing down the pinch-cock on the gas tubing so that the ignited vapors burn uniformly with the flame above the chimney but not above the wire bridge. Heat may be increased, if necessary, when the flame does not show above the chimney. The period of burning the vapors shall be 13 ± 1 min. If it is found impossible to meet the requirements for both flame and burning time, the requirement for burning time is the more important.

When the vapors cease to burn and no further blue smoke can be observed, readjust the burner and hold the heat as at the beginning so as to make the bottom and lower part of the sheet-iron crucible a cherry red and maintain for exactly 7 min. The total period of heating shall be 30 ± 2 min. which constitutes an additional limitation on the tolerances for the pre-ignition and burning periods. There should be no difficulty in carrying out the test exactly as directed with the gas burner of the type named, using city gas (about 550 B.t.u.) with the top of the burner about 2 in. below the bottom of the crucible. The time periods shall be observed with whatever burner and gas are used.

Remove the burner and allow the apparatus to cool until no smoke appears, and then remove the cover of the Skidmore crucible (about 15 min.). Remove the porcelain or silica crucible with heated tongs, place in the desiccator, cool, and weigh. Calculate the percentage of carbon residue on the original sample.

Special Procedure for Oils Having Carbon Residues in Excess of 5 Per Cent, Such as Heavy Crude Oils, Residuums, Heavy Fuel Oils, and Heavy Gas-enrichment Oils.—When the carbon residue as obtained by the procedure described (using a 10-g. sample) is in excess of 5 per cent, difficulties may be experienced due to boiling over of the sample. Trouble also may be encountered with samples of heavy products which are difficult to dehydrate. In the case, therefore, of samples showing by the usual method of test a carbon residue in excess of 5 per cent, the test shall be repeated in duplicate using a 5-g. sample weighted to the nearest 5 mg.

Special Procedure for Light Distillate Oils Having Carbon Residues Less Than 0.05 Per Cent, Such as No. 1 and No. 2 Fuel Oils.—In the case of these oils, the carbon residue test shall be made

on a 10 per cent residuum obtained by an adaptation of the distillation procedure in A.S.T.M. Designation: D 158, as follows:

To receive the distillate, use the 200-ml. graduate, without cleaning, used to measure the initial sample.

Maintain the condenser outlet at 32 to 40° F. (0 to 4.45° C.) throughout the distillation in the case of products having distillation end points below 600° F. (315° C.), and use the standard condenser temperatures 90 to 100° F. (32.2 to 37.8° C.) for products having end points above 600° F. (315° C.), or in those cases where waxy distillates are obtained.

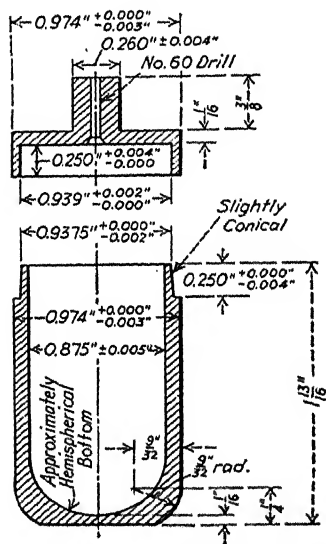
Carry out the distillation at the rate prescribed until exactly 178 ml. have been collected in the graduate, then discontinue the heating and allow the condenser to drain until 180 ml. (90 per cent of the charge to the flask) has been collected in the graduate. Replace the graduate with a small Erlenmeyer flask and catch any final drainage in this flask.

To this Erlenmeyer flask add, while still warm, the residue left in the distillation flask and shake well.

The contents of the Erlenmeyer flask then represent a 10 per cent residuum from the original product. While warm enough to flow freely, pour approximately 10 g. of the residuum into the weighed crucible to be used in the carbon residue test. After cooling, determine the weight of the sample accurately and carry out the carbon residue test in accordance with the procedure described. Report the percentage of carbon residue in the residuum as "carbon residue on 10 per cent residuum."

NOTE.—It is important that a clean distillation flask be used for each test.

Tests shall be run in duplicate and repeated if necessary until the percentages of carbon residue differ by not more than 10 per



Courtesy A.S.T.M.

FIG. 320.—Bulb for Ramsbottom Carbon Residue Test.

cent from an average. In the case of the carbon residue on 10 per cent residuum, duplicate determinations should differ by not more than 20 per cent from an average if the value is 0.10 per cent or higher. For lower values the deviations of individual determinations will be higher, increasing as the carbon residue decreases.

(II) *Ramsbottom Test for Carbon Residue*, which is conducted as follows:¹⁰⁰

This method of test outlines a procedure for determining the amount of carbon residue left on evaporating an oil under specified conditions, and is intended to throw some light on the relative carbon-forming propensities of oils. The results of the test must be considered in connection with other tests and the use for which the oil is intended.

The apparatus shall consist of the following:

(a) *Bulb Well*: A bulb well consisting of an iron tube with a flat closed end, open at the top, about 3 in. in height, 1 ± 0.005 in. in internal diameter, and having a wall thickness of about 0.05 in.

(b) *Coking Bulb*: A coking bulb, as illustrated in Fig. 320, made of stainless steel, consisting of a body with a hemispherical inner bottom and a flattened outer bottom, a friction-jointed cover terminating in a capillary, and having the following dimensions:

Dimensions of Bulb	Inches
Body	
Over-all height.....	1.8125
Outside diameter.....	0.971 to 0.974
Inside diameter.....	0.870 to 0.880
Outside diameter of relieved section.....	0.9375 to 0.9355
Height of relieved section.....	0.246 to 0.250
Radius of outside rounded bottom edge.....	0.281
Thickness of bottom.....	0.0625
Cap	
Over-all height.....	0.6875
Outside diameter.....	0.971 to 0.974
Inside diameter.....	0.939 to 0.941
Outside diameter of capillary.....	0.256 to 0.264
Height of capillary.....	0.375
Thickness of top.....	0.0625
Drill size of capillary.....	No. 60 Stubbs steel wire gage (0.04 in.)

(c) *Metal Bath*: A molten or solid metal bath with suitable arrangements for heating to a uniform temperature of $1,022^{\circ}$ F. In the case of the molten metal bath, means shall be provided for supporting the desired number of bulb wells immersed to a depth

of not less than 2.88 in. with the bottoms of the bulb wells approximately 1 in. from the bottom of the bath. In the case of the solid metal bath, holes 1 ± 0.005 in. in internal diameter and 3 in. in depth shall be drilled to accommodate the desired number of coking bulbs. The bottoms of these drilled bulb wells should be approximately 1 in. from the bottom of the bath.

(d) Temperature-measuring Devices: A thermocouple or other suitable temperature-indicating device located close to the bulb well with the lower end not less than 1 in. (25 mm.) from the bottom of the bath. It is desirable to protect the temperature-indicating device with a quartz or thin metal sheath when a molten bath is used.

Weigh a clean coking bulb dried in a desiccator, with cap removed but placed beside it on the balance pan, to the nearest 0.1 mg. Introduce the sample of oil (Note 1) into the bulb, and reweigh the bulb and cap, care being taken that no oil remains on the outside of the bulb. Place the cap firmly on the bulb and place the bulb assembly containing the sample in the bulb well in the bath, maintained at a temperature of $1,022^\circ \pm 9^\circ \text{F.}$, and allow to remain for 20 min. If there is any loss of oil from frothing, the test shall be discarded (Note 2). After removal, cool the bulb in a desiccator, examine to make sure there are no metal particles adhering to it, and weigh (Note 3).

NOTE 1.—The size of the sample should vary with the Ramsbottom carbon residue of the oil as shown in the following tabulation:

Ramsbottom Carbon Residue, per cent	Size of Sample, g.
Less than 3.....	4.0 ± 0.5
3 to 6.....	2.0 ± 0.4
Over 6.....	1.0 ± 0.3

NOTE 2.—Frothing may be due to water, which may be removed by heating if necessary.

NOTE 3.—After the test has been completed, the coking bulb may be cleaned for another test by scouring with steel wool, washing with naphtha, and drying in an oven at 220 to 230°F.

Special Procedure for Light Distillate Oils Having Ramsbottom Carbon Residues Less Than 0.05 Per Cent, Such as No. 1 and No. 2 Fuel Oils.—In the case of these oils, the Ramsbottom carbon residue test shall be made on a 10 per cent residuum obtained by an adaptation of the distillation procedure in A.S.T.M. Designation: D 158, as follows:

To receive the distillate, use the 200-ml. graduate, without cleaning, used to measure the initial sample.

Maintain the condenser outlet at 32 to 40° F. throughout the distillation, in the case of products having distillation end points below 600° F., and use the standard condenser temperatures of 90 to 100° F. for products having end points above 600° F., or in those cases where waxy distillates are obtained.

Carry out the distillation at the rate prescribed until exactly 178 ml. have been collected in the graduate, then discontinue the heating and allow the condenser to drain until 180 ml. (90 per cent of the charge to the flask) have been collected in the graduate. Replace the graduate with a small Erlenmeyer flask and catch any final drainage in this flask.

To this Erlenmeyer flask add, while still warm, the residue left in the distillation flask and shake well.

The contents of the Erlenmeyer flask then represent a 10 per cent residuum from the original product. While warm enough to flow freely, pour approximately 4 g. of the residuum into the weighed bulb to be used in the Ramsbottom carbon residue test. After cooling, determine the weight of the sample accurately and carry out the Ramsbottom carbon residue test in accordance with the procedure described. Report the percentage of carbon residue in the residuum as "Ramsbottom carbon residue on 10 per cent residuum."

The difference in weight of the bulb before and after the test, divided by the weight of the sample and multiplied by 100 is the Ramsbottom carbon residue expressed as a percentage of the oil sample.

Duplicate results should not differ by more than the amounts shown in the following table:

Deviation from the Average, per cent		
Ramsbottom Carbon Residue, per cent	Individual Results in One Laboratory	Average of Three Results Each in Different Laboratories
10	3	5
1	7	10
0.5	10	20
0.1	20	30

The relation between the Conradson carbon residue as determined by A.S.T.M. Method D 189 and the Ramsbottom carbon residue as determined by Method D 524 is given by the following equations:

$$3.8C = R^2 + 4.25R - 0.35 \quad (1)$$

$$65C = -R^2 + 90R + 10 \quad (2)$$

where C = Conradson carbon residue, and

R = Ramsbottom carbon residue.

Equation 1 applies to the range from 0.1 to 1.5 per cent Ramsbottom carbon residue or from 0.02 to 2.2 per cent Conradson carbon residue. Equation 1 should not be used for Ramsbottom values below 0.1 per cent as the Conradson values become negative for Ramsbottom values below 0.08 per cent.

Equation 2 applies to the range from 1.4 to 12 per cent Ramsbottom carbon residue or from 2.05 to 15 per cent Conradson carbon residue.

Table CXXXVIII shows the numerical relationships between Ramsbottom and Conradson carbon residue values. It is believed

TABLE CXXXVIII

AVERAGE RELATION BETWEEN RAMSBOTTOM AND CONRADSON CARBON RESIDUES, PER CENT

Ramsbottom	Conradson	Ramsbottom	Conradson	Ramsbottom	Conradson
0.08.....	0.00	1.20.....	1.63	4.00.....	5.44
0.10.....	0.02	1.30.....	1.80	4.50.....	6.08
0.20.....	0.14	1.40.....	1.99	5.00.....	6.70
0.30.....	0.27	1.50.....	2.20	6.00.....	7.90
0.40.....	0.40	1.60.....	2.33	7.00.....	9.10
0.50.....	0.535	1.70.....	2.46	8.00.....	10.25
0.60.....	0.675	1.80.....	2.60	9.00.....	11.35
0.70.....	0.82	1.90.....	2.73	10.00.....	12.43
0.80.....	0.97	2.00.....	2.86	11.00.....	13.50
0.90.....	1.13	2.50.....	3.52	12.00.....	14.55
1.00.....	1.29	3.00.....	4.17	13.00.....	15.60
1.10.....	1.45	3.50.....	4.81	14.00.....	16.50

that this correlation is dependable within the combined permissible deviations from the average as specified in the two methods.

(III) *Test for Carbon Residue in Paraffin Wax.* A test has also been standardized applicable to paraffin wax.¹⁰⁷

(D) SOLUBILITY TESTS

Test 21. **Solubility in Carbon Disulfide.** This test¹⁰⁸ is useful for purposes of identification, for ascertaining the adaptability of a bituminous substance for a given purpose, for gauging its uniformity of supply, and as a criterion of its quality (i.e., purity) and consequently its intrinsic value. Crude bituminous materials are often purchased on the basis of the percentage soluble in carbon disulfide. The presence of non-mineral matter insoluble in carbon disulfide may be regarded as just that much inert material, and in certain cases as an indication that the material has been carelessly prepared or overheated in its process of manufacture. The mineral matter may be regarded as an adulterant. In the case of native asphalts, the larger the percentage soluble in carbon disulfide, the greater will be their intrinsic value. The percentage and composition of the mineral matter will often indicate the source of the native asphalts. Asphalts derived from petroleum are substantially free from mineral constituents, and with the possible exception of the harder grades, contain little to no non-mineral matter insoluble in carbon disulfide.

With a native asphalt containing over 10 per cent of mineral matter, it is necessary to separate the portion soluble in carbon disulfide, before ascertaining its physical characteristics, fusing-point, and sometimes fixed carbon, in which case the soluble constituents should be recovered as will be described.

Two methods will be considered, depending upon whether or not the constituents are to be subjected to a detailed analysis.

Test 21a. Where the Constituents Are Not to Be Examined Further. In this case the method recommended follows along the lines of the one standardized¹⁰⁹ which is substantially as follows, differing however somewhat in phraseology: The sample shall be representative and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still as described in Test 25, the water-free distillate being returned to the residue. If

the material is hard and brittle, it may be ground * and dried at a temperature below the temperature of volatilization of the material, in a shallow nickel or iron dish.

A Gooch crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm. shall be set in the filter tube inserted in the stopper of the filtering flask. The flask shall be connected with the suction pump. Before suction shall be applied, the crucible shall be filled with asbestos † suspended in water which shall be allowed to settle partly in the crucible.

Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in water, allow it partly to settle in the crucible, and apply a light suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat is built up that weighs 0.5 ± 0.1 g. after ignition (Note). Wash the asbestos mat thoroughly with water, dry in the oven and ignite over a Bunsen burner. Cool the crucible in the desiccator, weigh, and replace it in the dry filter tube supported in the clean, dry filtering flask.

NOTE.—In the determination, the asbestos apparently adsorbs irreversibly a small amount of soluble bitumen (usually 1 to 5 mg. per gram of asbestos) which is not removed by subsequent washing with solvent. The weight of asbestos used, therefore, should be kept within the specified limits to insure reproducible results.

In the case of certain native asphalts containing finely divided mineral matter, the mineral residue is not easily retained by the filter and it is necessary to use method *II*, in order to obtain accurate results. Method *II* shall not be used unless the filter clogs unduly or unless the mineral matter passing through the filter exceeds 0.5 per cent.

I. Method Used Where But Little Finely-divided Insoluble Matter Is Present. Weigh approximately 2 g. of the sample into a tared conical flask, and add 100 ml. of C.P. carbon disulfide to

* Where it is not desirable to crush the rock or sand grains, a lump should be placed in the drying oven until it is heated through and softened at the lowest possible temperature, whereupon it may be crushed into a thin layer and dried as described.

† Asbestos (amphibole) cut in pieces not exceeding 1 cm. in length, shredded and shaken up with water.

the flask in small portions with continued agitation until all lumps disappear and nothing adheres to the bottom of the flask. Then stopper the flask and set aside for 15 min.

Decant the carbon disulfide solution carefully through the asbestos mat in the prepared Gooch crucible, with or without light suction as may be necessary, retaining as much of the sediment as possible in the conical flask until the solution has drained through the mat. With a small amount of carbon disulfide wash down the sides of the flask and transfer the sediment and precipitate from the flask to the asbestos mat. The flask may be scrubbed with a feather if necessary in order to remove all of the precipitate. Wash the contents of the crucible with carbon disulfide until the washings are colorless, then apply suction to remove the carbon disulfide. Dry the crucible in the oven at 100 to 125° C. for 20 min., cool in the desiccator, and weigh. The increase in weight over the original weight is the weight of matter insoluble in carbon disulfide. Ignite the crucible at a red heat and after thorough ignition, cool, and weigh. The weight of the insoluble matter after ignition is the weight of ash.

If insoluble matter adheres to the flask, dry the flask, and weigh. Add the increase in weight over the original weight to the weight of insoluble matter in the Gooch crucible.

In case there is any question involving the amount of mineral matter that may have passed through the filter, evaporate the filtrate, and burn the bituminous residue. If a carbonate mineral is present in the filtrate ash, add to the ash a few drops of ammonium carbonate solution, and dry at 100° C., then heat for a few minutes to a dull red heat, and cool in the desiccator. Weigh, and add the weight of ash obtained to the weight of matter insoluble in carbon disulfide.

NOTE.—In the event that water-soluble salts which are insoluble in carbon disulfide are present, the amount of these salts may be determined.

The following résumé embodies the calculations to be made:

The weight of the residue in the crucible shall be recorded. . . (1)

Ignite the residue in the crucible at a red heat to a clean ash, cool, add a few drops of ammonium carbonate solution, re-ignite to a low red heat, cool and weigh the residue. (2)

In case any insoluble matter adheres to the flask, it shall be dried, weighed and the increase over the original weight recorded. . . (3)

The filtrate containing the soluble matter shall be evaporated, the bituminous residue burned, and the weight of ash recorded. . (4)

Constituents Soluble in Carbon Disulfide equal the weight of material (dry) taken for analysis, minus the sum of items (1), (3) and (4). Calculate this in per cent.

Non-Mineral Constituents Insoluble in Carbon Disulfide equal item (1), minus (2), plus item (3). Calculate this in per cent.

Mineral Constituents equal the sum of items (2) and (4). Calculate this in per cent.

II. Method Used Where a Substantial Quantity of Finely-divided Insoluble Matter Is Present. Weigh approximately 2 g. of the sample into a tared conical flask, and treat with 100 ml. of C.P. carbon disulfide. Stopper the flask loosely, and shake at intervals until all large particles have been broken down. Then allow the flask to remain undisturbed for 48 hr.

Decant the solution into a similar tared flask, taking care to disturb as little of the residue as possible. Treat the residue in the first flask with 100 ml. of C.P. carbon disulfide, shake thoroughly, and allow both flasks to remain undisturbed for 48 hr.

Carefully decant the solution from the second flask upon the prepared Gooch crucible, filtering without the use of suction. Then in a similar manner filter the solution from the first flask. Wash the filter with fresh carbon disulfide. Add carbon disulfide to the residue remaining in each flask, shake thoroughly, and allow to settle for 24 hrs.

Decant the solutions from both flasks through the filter. Again wash the residues remaining in the flasks with carbon disulfide, decanting and filtering the solutions as before. Repeat this treatment until the washings are practically colorless. Maintain the temperature between 20 and 25° C. Dry the crucible and the flasks at 100 to 125° C., and weigh. The total increase in weight over the total original weight is the weight of matter insoluble in carbon disulfide. Evaporate the filtrate containing the bitumen, burn the bituminous residue, and weigh. Add the weight of ash thus obtained to the weight of insoluble matter in the Gooch crucible and conical flasks.

The sum of these weights is the total weight of matter insoluble in carbon disulfide.

The weight of sample taken calculated to a water-free basis minus the total weight of matter insoluble in carbon disulfide is the weight of bitumen. The percentages of bitumen and of ash shall be calculated on the basis of water-free material.

In every case the report shall indicate whether method *I* or *II* has been employed.

The author finds that in the presence of large quantities of finely-divided insoluble matter, the method may be materially shortened by adding a weighed quantity (about twice the weight of bituminous material) of freshly ignited, long-fibered amphibole to the bituminous substance in the first flask. On shaking with carbon disulfide, the asbestos serves to dilute the insoluble matter, preventing the latter from clogging the pores of the filter, and accordingly reducing the time of filtration.

A rapid colorimetric method depends upon the measurement of the amount of light by means of a photo-electric cell, which is transmitted through a solution of the bituminous substance, under controlled conditions, in comparison with a solution of known concentration. The solution is prepared by shaking 1 g. of the substance with a measured quantity of C.P. carbon disulfide for 20 minutes in a stoppered flask. A portion is then centrifuged to remove the insoluble constituents. The purified solution is poured into a hollow glass cell and subjected to a ray of light from a constant source. On closing the cell circuit, a microammeter in series with the photo-electric cell gives the reading of the soluble content upon a calibrated scale.* The scale is normally calibrated to give readings from 6 to 25 per cent, and the method is claimed to be accurate to within 0.1 per cent.

The following procedure has been standardized for determining inorganic matter or ash in solid, semi-solid or liquid bituminous substances: ^{199a}

The apparatus shall consist of the following:

(a) Crucible, with cover, approximate capacity 50 to 100 ml. either platinum, porcelain or fused silica.

* The apparatus, known as the "Photo-Bitometer" is supplied by Hutchinson's Testing Apparatus, Ltd., Westminster, S.W. 1, London.

- (b) Gas burner.
- (c) Analytical balance, capacity 50 g., sensitive to 0.001 g.
- (d) Desiccator with ground glass cover.

The sample shall be representative of the material. If it contains more than 2.0 per cent water, it shall be dehydrated by distillation in accordance with A.S.T.M. Method D 370 before testing. If the material is hard and brittle, it may be ground and dried at a temperature below the volatilization of the bitumen.

Weigh a sample of the material (2 to 5 grams) to the nearest milligram in a tared crucible. Heat slowly to drive off the combustible material without spattering until the bitumen is ignited. Then continue heating only enough to maintain combustion. When all readily volatile material is burned, the free carbon shall be ignited with a strong flame or in a muffle furnace until all carbonaceous matter has disappeared.

Cool in a desiccator and weigh. The heating shall then be repeated until a constant weight is obtained. The weighing and handling of the crucible shall be in accordance with accepted quantitative technique.

When the residue from determination of bitumen is being used, the filtrate containing the bitumen shall be evaporated, the bitumen ignited and the weight of ash added to the weight of ash in the residue.

When carbonate materials are present, the ash shall be moistened with a few drops of ammonium carbonate solution, dried at 100° C. (212° F.) and heated to a dull red for a few minutes, then cooled and weighed.

The total weight of ash (including ash in the filtrate) divided by the weight of sample and multiplied by 100 gives the percentage of ash.

Test 21b. Where the Constituents Are to Be Examined Further. Extract 25 to 50 g. of the moisture-free bituminous substance with carbon disulfide as previously described, increasing the quantities of carbon disulfide proportionately. In certain cases an extractor described in Test 59 may be conveniently employed.

An alternate procedure consists in dissolving the substance in benzol or carbon disulfide and centrifuging at 3500 to 4000 r.p.m. The separated mineral matter is centrifuged a second time with

clean solvent, whereupon the mineral matter is completely recovered and is entirely free from soluble organic matter.²⁰⁰

Residue Insoluble in Carbon Disulfide. This contains the non-mineral constituents insoluble in carbon disulfide (e.g., insoluble bituminous matter, free carbon and vegetable matter derived from associated soil) together with the mineral constituents retained by the filter (e.g., clay with 2 molecules of water in chemical combination,* silica, silicates, calcium and magnesium carbonates, calcium sulfate with half a molecule of water in chemical combination, iron pyrites, etc.). Dry in an oven at 220° F. to constant weight and mix well to obtain a uniform sample. If the mineral matter is coarse, pulverize sufficient of the well-mixed material in a mortar to 200 mesh or finer for use in carrying out the examination described in sections (a) to (e) inclusive.

(a) To determine the water in chemical combination with the clay and calcium sulfate, heat 10 g. to bright redness in a stream of dry hydrogen or illuminating gas, in an electric combustion apparatus, catching the water in a tared calcium chloride bulb, which is reweighed at the end of the operation. The gain in weight represents the water which should be calculated in percentage.

(b) To determine the sulfur present in sulfides, weigh 25 g. into a small flask closed with a stopper carrying a thistle tube filled with hydrochloric acid and an outlet tube leading into a beaker of bromine water. Introduce the acid and boil until all the hydrogen sulfide is expelled into the bromine water, which serves to oxidize it into sulfuric acid. Expel the excess of bromine by boiling and precipitate with barium chloride in the usual manner. Calculate the percentage of sulfur present.

(c) To determine the carbon dioxide present in carbonates, weigh 1 g. into a Schroetter or Mohr alkalimeter and treat with hydrochloric acid in the usual manner. The loss in weight represents the sum of the carbon dioxide (liberated from the carbonates) and the hydrogen sulfide (liberated from the iron pyrites). Subtract the weight of hydrogen sulfide (ascertained in b) to arrive at the weight of carbon dioxide present, and calculate its percentage.

(d) To determine the non-mineral constituents insoluble in car-

* The water of hydration in clay is held tenaciously, for none is given up during one hour's heating at 293° C., although it is expelled on heating to redness.

bon disulfide,²⁰¹ weigh 5 g. into a porcelain dish, treat with an excess of concentrated hydrochloric acid, evaporate to dryness on a water bath and then dehydrate the silica by heating in an air-oven at 220° F. for two hours. Boil up with water, filter on a weighed Gooch crucible and wash thoroughly with boiling water until the filtrate no longer shows a reaction for calcium salts. Dry at 220° F. until no further loss occurs and then weigh the residue, which represents the non-mineral constituents insoluble in carbon disulfide, together with clay and its water of hydration, silica, silicates, calcium sulfate and its water of hydration, etc. Ignite to redness until all the carbonaceous matter is consumed and reweigh. The loss in weight represents the sum of the non-mineral constituents insoluble in carbon disulfide, also the total water of hydration (originally associated with the clay and calcium sulfate). Subtract the per cent water of hydration (ascertained in *a*) to arrive at the per cent non-mineral constituents insoluble in carbon disulfide.

(*e*) To determine the iron, aluminum, calcium, magnesium, silica, sulfates, etc., weigh 2.5 g. into a platinum crucible and ignite until all the carbonaceous matter is consumed. Then fuse with potassium-sodium-carbonate, dissolve the melt in water, acidify with hydrochloric acid, evaporate to dryness to dehydrate the silica, take up with boiling water, filter on a weigh Gooch crucible to separate the silica, which is then washed with boiling water until free from salts, ignited and weighed. The filtrate is diluted to exactly 250 ml. at 77° F. whereupon 200 ml. are drawn off, oxidized by boiling with nitric acid, and the iron and aluminum precipitated with an excess of ammonia, filtered, washed, ignited and weighed together as Fe_2O_3 and Al_2O_3 . Calcium, magnesium and SO_3 are then determined in the filtrate in the usual manner. The remaining 50 ml. of the original filtrate are then reduced with metallic zinc and sulfuric acid, and the iron titrated with $\text{N}/10$ KMnO_4 . The Al_2O_3 is then calculated by difference. The percentage of clay present in the original material is calculated from the following formula: $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$. Gypsum is calculated from the following formula: $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$. Potassium and sodium are determined in a separate portion.

Siliceous and calcareous fillers may be determined by means of normal hydrofluoric and 0.5-normal hydrochloric acids, respec-

tively.²⁰² Iron may be determined colorimetrically as $\text{Fe}(\text{CNS})_3$ upon digesting the bituminous substance directly with sulfuric acid.²⁰³

Constituents Extracted: This comprises the soluble bituminous constituents together with any chemically combined mineral constituents, e.g., iron present in certain asphalts²⁰⁴ and fatty-acid pitches, lead present in the sludge asphalts, copper present in certain fatty-acid pitches, etc.,* also any colloidal mineral constituents not retained by the asbestos filter (e.g., clay, silica, calcium and magnesium carbonates, etc.). Cool the solution to exactly 77°F. , measure its volume and then dilute with carbon disulfide at 77°F. to the next higher 10-ml. mark. Thoroughly agitate the liquid and with a pipette transfer an aliquot portion into a tared crucible or dish. Evaporate the solvent over a steam bath, then incinerate the residue and ignite at white heat to a clean ash, until no further loss in weight occurs. Moisten with ammonium carbonate solution, re-ignite at a low heat (not beyond incipient redness) and reweigh. The ash represents the anhydrous clay, silica, calcium and magnesium carbonates, together with the mineral constituents originally in chemical combination with the bituminous matters. Calculate the mineral matter associated with the bituminous constituents in total carbon disulfide extract. Note that any water of hydration originally combined with the clay is not ascertained by the foregoing procedure, but the amount present is usually so small that it may be disregarded without vitiating the results. From the weight of the residue insoluble in carbon disulfide, calculate the soluble bituminous constituents by difference. Evaporate an aliquot portion of the well-mixed carbon disulfide extract to exactly the calculated weight.

It has also been noted that a small percentage of asphaltic binder often remains adsorbed by the associated mineral aggregate and may not be entirely removed upon extraction with carbon disulfide, although pyridine was found to have a substantially greater solvent action—although even this solvent did not completely remove the asphalt binder.²⁰⁵

It has been found that when the solvent is expelled by heating

* It is contended that in natural rock asphalts, a portion of the mineral matter is chemically united with the asphalt in the form of salts of sulfonated acids.

over a water bath for one hour, the fusing-point is increased 2 to 3° C.; also that the retention of 1 to 2 per cent of very fine mineral matter does not change the characteristics of the recovered bituminous constituents to any appreciable extent.²⁰⁶

The following variations have been proposed for recovering the bituminous constituents in their unaltered state:

I. Evaporation at Atmospheric Pressure. From the weight of the extracted mineral matter, calculate the bituminous matter by difference, and evaporate the carbon disulfide extract to exactly this weight. This may be conveniently performed by distilling and condensing most of the carbon disulfide over an incandescent light or an electric stove in a large distillation retort connected with a condenser and receiving flask. The retort shall have a thermometer inserted, with the bottom of the bulb approximately 1 in. from the bottom of the retort. The concentrated solution is transferred to a tared dish, evaporated dry on a steam bath with constant stirring, which may be conveniently accomplished with a motor-driven agitator, and the last traces of solvent expelled in an oven at 105° C. until the residue attains the calculated weight.²⁰⁷

An alternate procedure consists in distilling the filtered extract (containing a calculated amount of soluble constituents equal to approximately 100 g.) to a bulk of about 150 ml., then transferring the solution to a spherical flask of 300 ml. capacity. The evaporation is completed on a water bath which is heated from 40° C. up to the boiling-point, at which it is maintained for 5 hours, during which period the contents are stirred at intervals with a tared glass rod. Finally the flask is heated in an electrically heated oil bath, from an initial temperature of 105° C. to a final temperature of 130° C. which is kept constant for a period ranging from 4 to 6 hours, during which the contents are kept stirred with the tared glass rod. The oil bath is kept agitated with a motor driven paddle, which likewise causes the flask to revolve. The time of heating in the oil bath may be determined by dissolving asphalt of known penetration in carbon disulfide and ascertaining the time required at 130° C. to bring its penetration back to its original figure.²⁰⁸

An alternate procedure consists in extracting the asphalt with C.P. benzol, removing any finely-divided mineral constituents by centrifuging, and then distilling the extract (containing 50 to 100 g.

asphalt) in a 500-ml. short-neck flask (similar in design to that specified in A.S.T.M. test D 20-30) carrying a thermometer placed with its bulb $\frac{1}{4}$ in. above the bottom of the flask. A small flame placed at the side of the flask is regulated, so that the distillation proceeds at the rate of 5-ml. per minute. Excessive foaming is caused to subside by placing a flame on the sides of the flask adjacent thereto. At 150-200° C. when the foaming has ceased, the heat is raised in 5 min. to 250° C. with asphalts of about 200 penetration, and to 300° C. with asphalts of about 100 penetration, whereupon the distillation is stopped immediately and the residue poured into a container and stirred until it cools. It is claimed that asphalts of varying physical properties may thus be recovered without material alteration.²⁰⁹

II. Evaporation Under Vacuum. The solvent is first removed by distillation as in the foregoing, until the temperature of the residue in the flask reaches 300° F. The distillation flask is then connected to a filter pump and the distillation continued under a vacuum of 180 mm. mercury, raising the temperature to 500° F. at the rate of 5 to 6° F. per minute. If the original penetration is greater than 150 at 77° F., the distillation temperature should not exceed 425° F. The residue in the flask should then be weighed.²¹⁰ This procedure will give satisfactory results, provided the bituminous substance does not contain any appreciable quantity of constituents volatile at 400° F.

The following modification is claimed to give very accurate results.²¹¹ Dissolve the substance in C.P. carbon disulfide previously dried by contact with CaCl_2 . The extract should contain 5 to 6 per cent of the soluble constituents, and not exceed 240 ml. in volume, which may be attained by evaporation or by dilution. A shallow, circular brass or nickel plate as illustrated in Fig. 321 is supported horizontally (using a spirit level) in a vacuum-desiccator. About 60 ml. of the extract are poured into the plate and distributed uniformly over its bottom, whereupon vacuum is applied to the desiccator (180 mm. mercury) and maintained for 1½ hours. This induces the evaporation of most of the carbon disulfide. Any bubbles or blisters in the residue are pricked with a knife, and the entire layer scratched with the point of the knife in criss-cross directions as illustrated, so as to break it up into ridges, thereby ex-

posing the interior of the mass. It is then evacuated another $\frac{1}{2}$ hour. The plate is then warmed for 10 to 20 minutes at 70° C. in an oven to soften the film of bituminous substance, which is thereupon scraped out with a spatula. Then another 60 ml. of the extract are introduced in the vessel and treated in exactly the same manner. This operation is repeated four times or until all of the original extract (240 ml.) has been evaporated. In this manner,

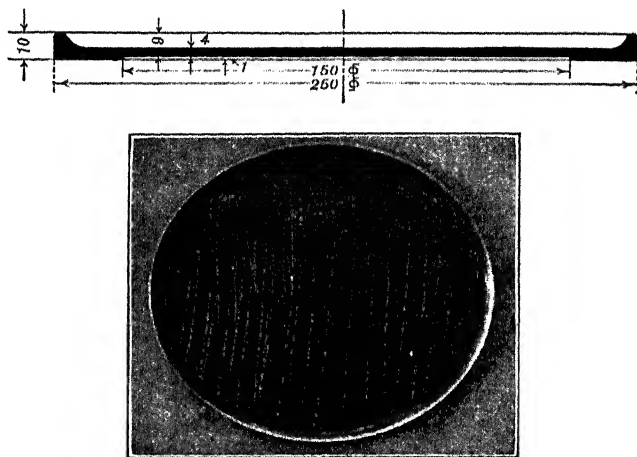


FIG. 321.—Shallow Plate for Evaporation.

about 15 to 16 g. of the asphalt is collected and utilized for further tests.

The following modification has been recommended for the recovery of liquid road asphalts:²¹²

With viscous cutbacks the thermometer reads 60° C. before the evel of the fluxing agent vapors (visible by mist formation) has risen to the side tube. In such cases heating is then continued until the level of the side tube has been reached, which may take place at a temperature of about 85° C.

With kerosine cutbacks the oil bath has to be heated to at most 250° C., with creosote cutbacks to 300° C. to recover the cutbacks. If cutbacks that have lost nearly all their fluxing agent are to be examined, the oil bath will be at a temperature of 300° C.

before the vapors reach the side tube. Distillation is then discontinued.

After cooling, the flask is rotated to distribute the condensed fluxing agent homogeneously in the residue. When this is done the binder is ready for further examination.

The distillation flask containing 600 g. of cutback is heated until the required temperature (Siegmann specifies 150 to 200° C. for gasoline; 200 to 220° C. for kerosine; 240° C. for gas oil) has been reached; then superheated steam of the same temperature is conducted through the mass in the flask. The distillation is continued until 10 times the amount of steam calculated on solvent (evaporation test) has passed over. It is of importance to ascertain whether, and how much, distillate passes over towards the end of the distillation. The best procedure is to plot a curve of the quantity of distillate against the quantity of water.

Though it is desirable to have an intake of 600 g. in order to make a reliable analysis of the separated solvent, in some cases, especially when analyzing road samples, the available quantity will be much smaller. In these cases the intake may be reduced to 300 g., a 1-liter distillation flask being used instead of a 2-liter flask. If the cutback contains only a small percentage of volatile products, the total quantity of steam (10 times the amount of distillate) would be somewhat on the low side. Therefore, the minimum quantity of steam should in any case be not less than 200 g.

The concentrated carbon disulfide extract is introduced into the flask of the Siegmann apparatus. The flask is placed in an oil bath, the temperature of which is raised carefully, so that the carbon disulfide passes over slowly (about 20 cc. per 15 minutes). When the greater part of the carbon disulfide has passed over, the apparatus is carefully subjected to vacuum, which is gradually increased to 40 cm. After the bulk of the carbon disulfide has been removed, the temperature of the oil bath is raised in 1 hour to 200° C.; upon continued heating the level of the fluxing agent is seen to rise slowly in the column. As soon as the vapors reach the bottom of the side tube of the fractionating column the vacuum is decreased, to prevent the fluxing agent from distilling over.

In the case of thinly liquid cutbacks, heating is continued until the thermometer reads 60° C., where the distillation is stopped.

A similar method may be adapted to bituminous emulsions, in which case it is modified as follows: About 35 g. of the emulsion are broken by adding a few drops of conc. HCl. The separated lumps are freed from acid by kneading in several portions of water with a glass rod. The asphalt is then dissolved in about 240 ml. of carbon disulfide, allowed to stand in a separatory-funnel until most of the water rises to the surface, whereupon the solution is drawn off into a flask and shaken repeatedly with granular CaCl_2 at 15 minute intervals. The carbon disulfide solution is then poured off and treated as described above to recover the dissolved asphalt.

III. Evaporation in a Stream of CO_2 . This method of test covers the procedure for the extraction of benzol soluble bitumen from asphaltic mixtures, the removal of mineral matter from the solution and the recovery of the bitumen from solution in sufficient quantity for further testing.

NOTE.—This method has been studied by the committee for asphalts harder than 150 penetration. Suitability of the method for softer residues has not been determined.

The apparatus shall consist of the following:

(a) Oven: An oven capable of maintaining the temperature at 210 to 220° F.

(b) Balance: A balance capable of weighing 5000 g.

(c) Extractor: An extraction apparatus as shown.

(d) Centrifuge: A centrifuge capable of handling two 8-oz. wide-mouth bottles at 770 times gravity.

(e) Bottles: A supply of 8-oz. wide-mouth bottles.

(f) Distillation Assembly: A distillation assembly as shown in Fig. 322 and consisting of the following items:

A heat-resistant glass distillation column 250 mm. in length and 25 mm. in diameter, and provided with a side arm 150 mm. in length and 8 in. in diameter; an iron tripod; a 6 by 6-in. 20-mesh wire gauze with an asbestos center; a gas burner; a water-jacketed condenser 475 to 500 mm. in length; three thermometers conforming to the requirements for thermometers as prescribed in A.S.T.M. Designation: E 1; a 250-ml. Erlenmeyer receiving flask and a 50-ml. Erlenmeyer filtering flask; a 500-ml. graduated cylinder; forks of assorted sizes; a ringstand and supports; a distillation flask as illustrated; an oil bath for the distillation flask; a gas-flow meter; a gas inlet coil; and a cylindrical, flat-bottom, seamless tin container of 6-oz. capacity. The container shall be 70 mm. (2¾ in.) in diameter and 45 mm. (1¾ in.) in depth (see Note).

NOTE.—Containers known in the drug trade as seamless "ointment boxes" may be obtained in the dimensions conforming to these requirements.

The reagents shall consist of benzol (thiophene free, dry, with a boiling range of 79.5 to 81.5° C.) and carbon dioxide.

A sample of sufficient size to result in at least 100 g. of recovered bitumen is required. About 1000 g. of sheet-asphalt mixtures will usually be sufficient unless the largest particles in the sample are 1 in., in which case 2000 g. will usually be required. Mixtures containing larger aggregates will require still larger samples.

The sample shall be placed in the oven at 210 to 220° F. for 15 min., broken into pieces and dried in the oven for an additional 30 min. The desired amount of the sample shall be weighed to the nearest 5 g. and placed in the basket of the extractor with the stirrer in place. The extractor shall be charged with 400 ml. of benzol and the wire cone hung on the bottom of the basket. The basket shall be inserted in the extractor, the condenser cover placed on the extractor and the handle placed on the stirrer. Cold water shall be circulated through the condenser. The electric heater shall be connected and the sample extracted until the benzol is colorless (see Note). When the sample contains a mineral filler, the stirrer shall be turned by hand one-half turn every 15 min. (after the benzol becomes straw colored) to break up the settled filler and remove the last traces of bitumen.

NOTE.—This can be observed by placing a light at one window of the extractor and observing the dripping benzol through the other window.

The benzol solution shall be drawn off and the volume increased to 400 ml. by the addition of fresh benzol, using this solution to wash the extractor if necessary. The solution shall be poured into two 8-oz. wide-mouthed bottles, balanced accurately, stoppered, and placed in the centrifuge. The solution shall be centrifuged at room temperature for 30 min. at 770 times gravity using the distance (in feet) from the center of the centrifuge to a point midway in the liquid, as the value of R in the following formula for calculation of speed of the centrifuge:

$$\text{Speed, r.p.m.} = \frac{1500}{\sqrt{R}}$$

The solution shall be poured into a previously weighed 500-ml., three-neck flask, care being taken not to disturb or include the sediment.

Using the distillation assembly shown, the temperature shall be raised inside the flask to 300° F. (148.9° C.) at such a rate that the benzol is collected at a rate of 50 to 70 drops per min. As soon as this rate falls off, carbon dioxide gas shall be admitted slowly, increasing to a rate of 800 to 900 ml. per min. The contents of the flask shall be maintained at 295 to 305° F. (146.1 to 151.7° C.) for exactly 30 min. with full carbon dioxide gas rate. The outside bath temperature ordinarily shall be held 15 to 25° F. (8 to 14° C.) higher than the inside in order to maintain a sample temperature of 295 to 305° F. (146.1 to 151.7° C.).

The flame shall be removed, the carbon dioxide gas shut off, and the apparatus disassembled. The outside of the flask shall be cleaned and the flask and contents weighed. The percentage of bitumen shall be determined by the following formula:

$$\text{Bitumen, per cent} = \frac{\text{wt. of flask and contents} - \text{wt. of flask}}{\text{wt. of original sample}} \times 100$$

The contents of the flask shall then be poured into the 6-oz. container and cooled.

A maximum temperature of 300° F. is recommended for asphalts softer than 100 penetration at 77° F.; likewise 350° F. for asphalts having a fusing-point of over 150° F. (R. and B.). It is important that the extracted asphalt solution be distilled on the same day that it is dissolved, otherwise considerable alteration may occur if the solution is allowed to stand for more than 10 hours, regardless of whether benzol or carbon disulfide is used, and will result in a change in the physical characteristics of the extracted asphalt.²¹³

Boiling trichloroethylene is claimed to give better results and to act more rapidly than the use of carbon disulfide in the cold.²¹⁴ It has also been suggested that the asphalt be first dissolved in chloroform, filtered, benzol added and the extraction continued in the usual manner.²¹⁵

The following procedure has been proposed for recovering asphalts from paving compositions:²¹⁶

The apparatus adopted for the recovery of penetration asphalts is shown in Fig. 323. The capacity of the flask is 1 liter, the capillary inlet is 1 mm. in inside diameter and reaches to within 3 mm. of the bottom of the flask, and the fractionating column is 25 cm. long and 8 mm. in inside diameter.

The asphalt solution, which as extracted usually contains 5 to 10 per cent of asphalt in carbon disulfide or benzene, is concen-

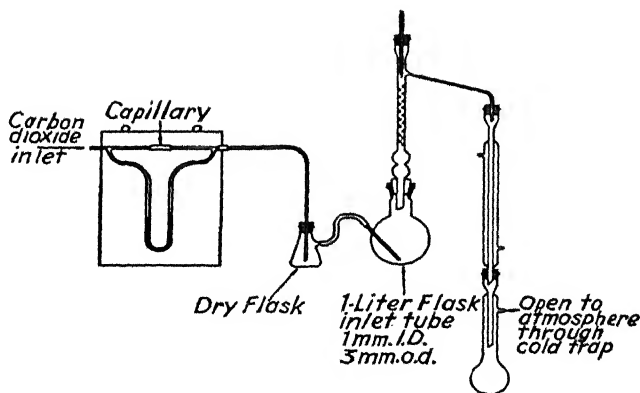


FIG. 323.—Apparatus for Extracting Asphalt from Paving Compositions.

rated without vacuum in a flask provided with a reflux column to a concentration of approximately 25 per cent. About 400 cc. of this solution are introduced into the apparatus (Fig. 323), a few silicon carbide boiling chips are added, and the solution is concentrated without vacuum or agitation over a water bath at 55 to 60° C. if carbon disulfide is the solvent; at 90 to 100° C. if benzene is the solvent. The bulk of the solvent is distilled under these conditions, the temperature being adjusted to maintain a distillation rate just short of a steady stream. When the temperature has reached 100° C. a previously heated oil bath is substituted for the water bath. The oil bath is then gradually heated to 150° C. when using either benzene or carbon disulfide and held at this temperature throughout the remainder of the recovery process.

After the distillation rate has dropped off to 5 to 10 drops per minute a stream of carbon dioxide gas is passed through the asphalt

mass for 30 minutes at the rate of 900 cc. per minute. If at the end of this time condensed oils are visible in the fractionating column, careful heating will cause them to flow back into the flask. The flask is finally rotated rapidly to remix any oils condensed on its upper surfaces, and the recovered asphalt is ready for analysis.

As a safety precaution a filter flask filled with solid carbon dioxide should be attached to the open end of the apparatus. This will prevent air being accidentally drawn back into the carbon disulfide or benzene system.

A modification of the procedure, consists in distilling off the solvent in a stream of CO_2 under reduced pressure.²¹⁷ The following method involving this procedure has been proposed:²¹⁸

The solution of asphalt in carbon disulfide is poured into a 500-ml. round-bottomed flask and heated in an oil bath to 120°C . The temperature is then raised to 150°C . over a period of 20 to 25 minutes and maintained at this temperature until the carbon disulfide distills at approximately 1 drop per second. Dry carbon dioxide is then introduced, slowly during the first minute, and then at the rate of 500 ml. per minute for 15 minutes. The pressure is thereupon reduced to 20 cm. of mercury and the flow of gas increased to 900 ml. per minute and maintained at this rate for 10 minutes. Finally, the pressure is slowly increased, the flow of gas then gradually decreased, the oil bath removed, and the asphalt residue poured into a receptacle for further examination. It has been observed that the presence of water during the recovery operation will result in a reduction of 10 to 15 per cent in the penetration of the asphalt. Hence in the case of road materials containing mineral constituents, the specimen should first be dehydrated, whereupon sufficient to yield about 100 g. of extract is dissolved in carbon disulfide, and the solution poured into centrifugal tubes and centrifuged at approximately 1,500 rpm. for 10 to 15 minutes. The supernatant liquid is carefully decanted into the recovery flask and treated without delay as described above.

It has also been noted that the true ductility of the extracted asphalt will be restored if the asphalt is heated to 300°C .²¹⁹

IV. Rapid Methods. A method for separating asphalt from limestones and dolomites consists of the following:²²⁰ Weigh out 10 g. of the rock asphalt and add 75 ml. of a mixture of 30 ml.

concentrated HCl (sp. gr. 1.19) and 45 ml. ethyl ether. Stir 10 minutes, until the carbonates dissolve; add 75 ml. water and warm over a water bath until the ether is expelled. Then filter and wash with warm water. Dry the filter at 105° C. and extract the filter with carbon disulfide to recover the asphalt. The extract may be treated by any of the procedures described above.

Another rapid method has also been proposed for ascertaining the asphalt present in sulfur deposits.²²¹

The soluble bituminous constituents, after being separated in their pure state, as previously described, may be then examined further for their physical and chemical characteristics. Due allowance should be made for the fact that any bituminous matter insoluble in carbon disulfide will be retained mechanically by the extracted mineral constituents, which with asphaltic products is usually relatively unimportant, but in the case of coal-tar products, will amount to a considerable item.

Test 22. Carbenes. The expression "carbenes" has been applied to that portion of bituminous substances soluble in carbon disulfide but insoluble in carbon tetrachloride. This term was originally proposed by Clifford Richardson.²²² The test is of value in identifying bituminous substances, gauging their uniformity of supply, for purposes of factory control, and as a criterion of their quality. Certain hard native asphalts and asphaltites, particularly grahamite, normally contain a percentage of carbenes, whereas petroleum asphalts do not show carbenes unless they are overheated or over-blown. If more than 0.5 per cent is present in petroleum asphalts, their quality is to be regarded as questionable. Carbenes are found in tars and pitches in varying amounts.²²³

Although carbenes are found in grahamite and certain hard natural asphalts when tested as such, they disappear upon fluxing to a softer consistency. With petroleum asphalts, tars and pitches, the carbenes are of a different character, since they are insoluble in fluxes and do not disappear upon being so treated.

This test is carried out by following the same procedure as in determining the solubility in carbon disulfide (Test 21), but replacing the latter with carbon tetrachloride. The carbon tetrachloride must be free from carbon disulfide, which may be insured by distilling it under a dephlegmator, discarding any distillate below

76° C. The solvent is then filtered through calcium chloride, and any free hydrochloric acid removed by blowing dry air through it.

The procedure has been standardized as follows:²²⁴

The apparatus shall consist of the following:

(a) Gooch Crucible, approximately 4.4 cm. in width at the top, tapering to 3.6 cm. at the bottom, with a depth of 2.5 cm.

(b) Asbestos (amphibole), Gooch grade, acid washed, cut in pieces not exceeding 1 cm. in length, shredded, and shaken up with water.

(c) Flasks: Two 125-ml. conical flasks such as Erlenmeyer flasks.

(d) Filtering Flask.

(e) Filter Tube.

(f) Section of Rubber Tubing, to hold the Gooch crucible on the filter tube.

(g) Drying Oven.

(h) Bunsen Burner.

(i) Suction Pump.

(j) Analytical Balance.

(k) Desiccator.

The sample shall be representative, and if it contains more than 2 per cent of water it shall be dehydrated by distillation in a copper still and the water-free distillate returned to the residue. If the material is hard and brittle, it may be ground, and dried at a temperature below the temperature of volatilization of the material.

Insert the filter tube in the stopper of the filtering flask, set the Gooch crucible in the filter tube, and connect the flask to the suction pump. Fill the crucible with some of the suspension of asbestos in water, allow it partly to settle in the crucible, and apply a light suction to draw off the water, leaving a firm mat of asbestos in the crucible. Add more suspended asbestos and repeat the process until a mat is built up that weighs 0.5 ± 0.1 g. after ignition (Note). Wash the asbestos mat thoroughly with water, dry in the oven, and ignite over a Bunsen burner. Cool the crucible in the desiccator, weigh, and replace it in the dry filter tube supported in the clean, dry filtering flask.

NOTE.—In the determination, the asbestos apparently adsorbs irreversibly a small amount of soluble bitumen (usually 1 to 5 mg. per gram of asbestos) which is not re-

moved by subsequent washing with solvent. The weight of asbestos used, therefore, should be kept within the specified limits to ensure reproducible results.

Weigh approximately 2 g. of the sample into a tared conical flask, and add in small portions 100 ml. of C.P. carbon tetrachloride with continued agitation until all lumps disappear and nothing adheres to the bottom of the flask. Then stopper the flask and set aside in subdued light for at least 12 hrs.

Decant the carbon tetrachloride solution carefully through the asbestos mat in the prepared Gooch crucible, with or without light suction as may be found necessary, retaining as much of the sediment as possible in the conical flask until the solution has drained through the mat. With a small amount of carbon tetrachloride wash down the sides of the flask and transfer the sediment and precipitate from the flask to the asbestos mat. The flask may be scrubbed with a feather, if necessary, in order to remove all of the precipitate. Wash the contents of the crucible with carbon tetrachloride until the washings are colorless, then apply suction to remove the carbon tetrachloride. Dry the crucible in the oven at 100 to 125° C. for 20 min., cool in the desiccator, and weigh. The increase in weight over the original weight is the weight of matter insoluble in carbon tetrachloride.

If insoluble matter adheres to the flask, dry the flask, and weigh. Add the increase in weight over the original weight to the weight of insoluble matter in the Gooch crucible.

In case there is any question involving the amount of mineral matter that may have passed through the filter, evaporate the filtrate, and burn the bituminous residue. If a carbonate mineral is present in the filtrate ash, add to the ash a few drops of ammonium carbonate solution, and dry at 100° C., then heat for a few minutes to a dull red heat, and cool in the desiccator. Weigh, and add the weight of ash obtained to the weight of matter insoluble in carbon tetrachloride.

The weight of sample taken calculated to a water-free basis minus the total weight of matter insoluble in carbon tetrachloride is the weight of bitumen soluble in carbon tetrachloride. The percentage shall be calculated on the basis of water-free material.

The proportion of bitumen soluble in carbon tetrachloride shall be reported on the basis of total bitumen taken as 100, as follows:

$$P = \frac{\text{Bitumen soluble in carbon tetrachloride}}{\text{Total bitumen}}$$

where P = proportion of bitumen soluble in carbon tetrachloride.

The difference between the percentages soluble in carbon disulfide and carbon tetrachloride, respectively, represents the per cent of "carbenes."

Test 23. Solubility in Petroleum Solvents. This test is employed mainly for purposes of identification. It is also used to a certain extent for determining the adaptability of bituminous substance for a given use, for gauging the uniformity of supply, and for purposes of factory control. As a general principle, the harder the bituminous product, the smaller will be the percentage that dissolves in petroleum naphtha. Asphaltites are relatively insoluble in this menstruum. Mineral waxes, peat-, lignite- and shale-tars or pitches are largely soluble. The solubility of native and petroleum asphalts varies, depending largely upon their hardness, and also in the case of petroleum asphalts upon the extent to which the distillation has been driven. Coal-tar pitches are relatively insoluble in petroleum naphtha.

I. Solubility in Petroleum Naphtha. The portion soluble in petroleum solvents has been termed "petrolenes" by some, and "malthenes" by others, whereas the nonmineral constituents remaining insoluble are generally referred to as "asphaltenes."²²⁵ Asphaltenes are the characteristic constituent of asphalt, which serve to distinguish it from all other petroleum products, and are responsible for its plastic properties.²²⁶

It is important that the petroleum naphtha should be derived from petroleum composed entirely of open-chain hydrocarbons, and test exactly 88° Baumé, equivalent to a specific gravity of 0.638 at 60° F./60° F. At least 85 per cent by volume should distil between 95 and 150° F. The density and character of the naphtha is important, since heavy distillates or products derived from petroleum containing unsaturated or cyclic hydrocarbons, will exert a greater solvent action upon the bituminous substance.

The results will be more consistent if the petroleum naphtha is first washed with fuming sulfuric acid to remove the aromatic constituents. There appears to be no difference in the results if the

precipitation is carried out at any temperature between 0 and 32° C. As the boiling-point of the petroleum spirits employed in making the test increases, the quantity of precipitate decreases. The fraction below 105° F. appears to give the most reliable results.²²⁷ The following method has been standardized:²²⁸

This determination is made in the same general manner as the total bitumen determination, except that 100 cc. of 86 to 88° Baumé paraffin naphtha, at least 85 per cent distilling between 35 to 65° C. is employed as a solvent instead of carbon disulfide. Considerable difficulty is sometimes experienced in breaking up some of the heavy semi-solid bitumens; the surface of the material is attacked, but it is necessary to remove some of the insoluble matter in order to expose fresh material to the action of the solvent. It is therefore advisable to heat the sample after it is weighed, allowing it to cool in a thin layer around the lower part of the flask. If difficulty is still experienced in dissolving the material, a rounded glass rod will be found convenient for breaking up the undissolved particles. Not more than one-half of the total amount of naphtha required should be used until the sample is entirely broken up. The balance of the 100 cc. is then added, and the flask is twirled a moment in order to mix the contents thoroughly, after which it is corked and set aside for 30 minutes.

In making the filtration, the utmost care should be exercised to avoid stirring up any of the precipitate, in order that the filter may not be clogged and that the first decantation may be as complete as possible. The sides of the flask should then be quickly washed down with naphtha and, when the crucible has drained, the bulk of insoluble matter is brought upon the felt. Suction may be applied when the filtration by gravity almost ceases, but should be used sparingly, as it tends to clog the filter by packing the precipitate too tightly. The material on the felt should never be allowed to run entirely dry until the washing is completed, as shown by the colorless filtrate. When considerable insoluble matter adheres to the flask, no attempt should be made to remove it completely. In such cases the adhering material is merely washed until free from soluble matter and the flask is dried with the crucible at 100° C. for about 1 hour, after which it is cooled and weighed. The percentage

of bitumen insoluble is reported upon the basis of total bitumen taken as 100.

The difference between the material insoluble in carbon disulfide and in the naphtha is the bitumen insoluble in the latter. Thus, if in a certain instance it is found that the material insoluble in carbon disulfide amounts to 1 per cent and that 10.9 per cent is insoluble in naphtha, the percentage of bitumen insoluble would be calculated as follows:

$$\frac{\text{Bitumen insoluble in naphtha}}{\text{Total bitumen}} = \frac{10.9 - 1}{100 - 1} = \frac{9.9}{99} = 10 \text{ per cent}$$

NOTE.—When it is necessary to make an ash correction and recarbonation of the ash, procedure as given under Test 21a shall be used.

Asphaltenes show increasing solubility in solvents in the order of their surface tension (e.g., ether, benzol, carbon disulfide and pyridine). Ether has been recommended as a substitute for petroleum naphtha, because it is a homogenous substance, not requiring standardization, and in addition has good flocculating properties and exerts a greater solvent action on hydroxy acids present in certain asphalts.²²⁹

II. Solubility in Pentane, Hexane, etc. Solvents of definite chemical composition have been proposed from time to time as substitutes for the rather variable petroleum naphtha, including: normal-pentane (C_5H_{12}), normal-hexane (C_6H_{14}), iso-pentane (C_5H_{12}),²⁸⁰ ethyl ether,²⁸¹ the use of chloroform at elevated temperatures,²⁸² etc. It should be noted, however, that the yield of asphaltenes will depend upon the particular solvent employed. Thus, pentane-asphaltenes will run about 8 per cent higher than ether-asphaltenes. Similarly, when ethyl ether is used, the asphaltenes will run less than with petroleum naphtha (i.e., 20–24 per cent against 35–38 per cent). It has been proposed to apply the term “difference asphaltenes” to the portion soluble in ethyl ether, but insoluble in petroleum naphtha. In Trinidad Lake asphalt, the oxygen compounds appear to be concentrated in the “difference asphaltenes,” whereas the sulfur compounds are largely present in the ether-asphaltenes.

Cyclo-hexane has been proposed for identifying cracked asphalts as well as for detecting the presence of decomposition prod-

ucts formed during treatment, as in the case of blowing, whereupon the cyclo-hexane insoluble constituents are increased.²³³

The percentage of asphaltenes varies considerably; thus, with asphalts all having the same R. and B. fusing-point of 140° F., the following are obtained: extracted asphalt from Trinidad asphalt 37 per cent, Mexican residual asphalt 20 per cent, California residual asphalt 12 per cent, Colombian residual asphalt 16 per cent, Illinois residual asphalt 12 per cent, Texas residual asphalt 9 to 17 per cent.²³⁴

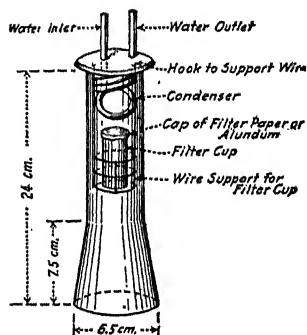
Test 24. Insoluble in Benzol ("Free Carbon"). This test is generally used for testing tars and pitches for the presence of non-mineral matter insoluble in hot benzol or carbon disulfide. The test is of value for purposes of identification, for ascertaining the adaptability of the tar or pitch for a given purpose, and for gauging its uniformity of supply. Tars and pitches containing large percentages of insoluble matter, known as "free carbon," are objectionable for certain manufacturing purposes, since the free carbon acts as so much inert matter. The term "free carbon" is a misnomer, since it is not elemental carbon, but a complex mixture of hydrocarbons of high molecular weight, containing 90.0 to 91.7 per cent carbon, 3.4 to 4.0 per cent hydrogen, 1.0 to 1.2 per cent nitrogen, 2.5 to 3.3 per cent oxygen and 0.7 to 1.4 per cent sulfur, on the ash-free basis.²³⁵ The presence of hydrogen has been explained by the great adsorptive power of carbon in its pure state, which retains hydrocarbons tenaciously, as well as hydrogen, which is not driven off at temperatures as high as 800° C. Free carbon is more soluble in aniline or pyridine than in benzol or carbon disulfide. It has been found that "heavy oils" derived from coal tar on distillation (270–320° C.), quinoline and phenyl nitrite are the most effective solvents. However, with the less effective solvents, the "free carbon" was increased by prolonged contact before filtration, also by exposure to daylight.²³⁶ Selenium oxychloride exerts the greatest solvent action, but unfortunately the residue cannot be freed from this solvent. Free carbon is also partially decomposed by digesting with hot fuming nitric acid.²³⁷

Marcusson has found that the benzol-insoluble constituents of vertical- and horizontal-retort coal-tars, amounting to 7 per cent and 24 per cent respectively, consisted of oxy-acids 8.6 per cent and

0.5 per cent; pyridine soluble resins 73 per cent and 16.3 per cent; pyridine insoluble resins 18.4 per cent and partly coked material 0 per cent and 51.2 per cent respectively.²⁸⁸

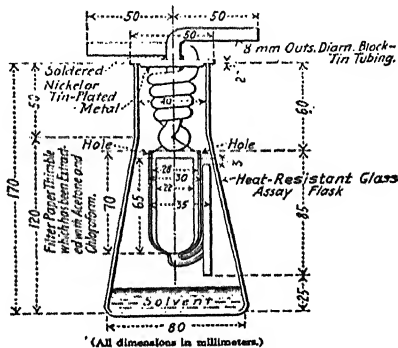
The following method of extraction has been standardized:²⁸⁹ Tars must be dehydrated before extracting, and pitches, if sufficiently hard, ground to a powder.

(I) *Porous Thimble Method*: The extractor shall be of a form shown in Fig. 324, or of a similar form in which the substance



Courtesy A.S.T.M.

FIG. 324.—Alundum Thimble Extraction Apparatus.



Courtesy A.S.T.M.

FIG. 325.—Paper Thimble Extraction Apparatus

is subjected to direct washing by the vapors of the boiling solvent. The filtering medium shall be a flat-bottom, 30 by 80 mm. RA-98 alundum thimble. The thimble shall be suspended in the extraction flask either by a wire basket hung from two small hooks on the under surface of the metal cover of the flask, or it shall be supported by making perforations near the upper edge of the thimble and suspending from the cover by German silver or platinum wire.

An amount of material which shall contain 10.0 g. \pm 0.1 g. of the substance shall be weighed into a 100-ml. beaker; 50 ml. of pure benzol shall be added and the solution stirred thoroughly. The solution shall be transferred at once to the weighed alundum thimble. The beaker shall be rinsed clean with pure benzol and the washings added to the thimble. The thimble shall then be covered with a lid of alundum ware and placed immediately in the extraction apparatus. The extractor shall contain a suitable quantity of pure

benzol and shall be heated sufficiently to boil the solvent. The extraction shall be continued until the solvent descending from the thimble is colorless. The thimble shall then be dried at $105^{\circ}\text{C.} \pm 5^{\circ}\text{C.}$, cooled in a desiccator, and weighed.

(II) *Asbestos Mat Method*: The filtering medium shall consist of a No. 3 Gooch, Coors porcelain or equivalent crucible approximately 3.5 cm. in diameter at the top, tapering to 2.2 cm. at the bottom with a depth of 4.0 cm., containing a mat of acid-washed medium fiber asbestos approximately 2 mm. in thickness.

The Gooch crucible shall be placed in the suction apparatus and filled with acid-washed medium fiber asbestos suspended in water. Gentle suction shall be applied and more of the suspension, if necessary, shall be added to make a mat approximately 2 mm. in thickness. With the suction still on, the pad shall be washed with water until all small particles of asbestos are removed. The crucible shall be dried at $105^{\circ}\text{C.} \pm 5^{\circ}\text{C.}$, placed in a desiccator, and weighed.

An amount of material which shall contain 10.0 g. \pm 0.1 g. of the substance shall be weighed into a 125-ml. Erlenmeyer flask; 50 ml. of pure benzol shall be added and the solution stirred thoroughly and brought to a boil. The hot solution shall be carefully poured into the weighed prepared Gooch crucible without suction until the mat is covered. Then gentle suction shall be applied to the crucible and the remaining solution added, taking care that the mat is covered with solution at all times. The Erlenmeyer flask shall be rinsed clean with pure benzol and the washings added to the crucible. Benzol shall be added to the crucible until the descending solvent is colorless. The crucible shall then be dried at $105^{\circ}\text{C.} \pm 5^{\circ}\text{C.}$, cooled in a desiccator, and weighed.

(III) *Paper Thimble or Filter-paper Method*: A paper thimble or else two thicknesses of S. & S. No. 575 or Whatman No. 5 hardened filter-paper (15 cm. in diameter, arranged in cup shape by folding) may be used in connection with an extractor of the form shown in Fig. 325.²⁴⁰ In this case the procedure is similar to that outlined in the Porous Thimble Method (I).

It is recommended that a ground-glass stopper with a flat or a mushroom-type top be inverted in the bottom of the extractor, to support the thimble, and thereby prevent obstructing the syphon.²⁴¹

The insoluble residue of "free carbon" includes the mineral ash, which may be ascertained by incineration. Some tars (e.g., blast-furnace tars) contain substantial amounts of ash, which would otherwise introduce serious errors. It has been found that the amount of free carbon will increase with the time the solvent remains in contact with the tar or pitch. The amount of such increase varies with the particular solvent employed. In the case of carbon disulfide or chloroform, the increase is less than with benzol or toluol (or mixtures of these two) and the amount apparently reaches a maximum in about 120 hours.²⁴²

A method has been proposed in which 5 g. of the pitch is heated with 200 ml. tetralin in an autoclave at 240–250° C. under a pressure of 12–13 atmospheres for 2 hours. Upon cooling, the liquid is filtered through a Gooch crucible and washed successively with 50 ml. tetralin and 100 ml. benzol. The residue is dried for 2 hours at 150° C. in an atmosphere of CO₂ and weighed.²⁴³

Other solvents that have been proposed from time to time for determining the free-carbon content of tars and pitches, include: toluol,²⁴⁴ a mixture of benzol and toluol,²⁴⁵ xylol,²⁴⁶ aniline,²⁴⁷ pyridine,²⁴⁸ tetrahydronaphthalene,²⁴⁹ nitrobenzol, carbon disulfide,²⁵⁰ etc. The quantity of insoluble matter ("free-carbon") recovered, depends upon the following variables: (1) the type of solvent used, (2) the ratio of solvent to sample extracted, (3) the temperature at which the test is conducted, and (4) the time of contact with the solvent used. It has also been reported that the physical properties of the recovered bituminous constituents are altered least when benzol is used, carbon disulfide comes next in merit, whereas the chlorinated hydrocarbons cause considerable hardening.²⁵¹ The chlorinated solvents in the sequence of their decreasing hardening effects are: carbon tetrachloride, trichloroethylene, dichloroethylene and chloroform.²⁵²

Anthracene oil (freed from crystallizable substances at 15° C.) has also been proposed, in which case, 5 g. of the substance are digested with 5 g. anthracene oil under a reflux condenser for 4 to 5 hours, cooled, diluted with 500 ml. benzol, filtered, washed with benzol, etc., as in the preceding.²⁵³ Aniline has also been suggested as extracting medium, involving the digestion of 5 g. of substance with 25 ml. aniline over a steam bath for ½ hour, pouring the

liquid on a porous clay plate to absorb the solvent, washing the residue with pyridine, followed by benzol, drying and weighing.

(IV) *Filtration Method*: An ingenious method has been described²⁵⁴ for actually filtering the free carbon from liquid tars as follows:

A specially constructed pressure filter submerged in a hot water bath was used (Fig. 326). The filter medium, *E*, consisted of

an alundum extraction shell of medium porosity (Norton RH 360-34 mm. diameter, 100 mm. high), which was cemented into the brass collar, *C*, by means of a paste of zinc oxide in water glass. The collar fitted snugly between the upper and the lower part of the outer cylinder, *O*. Lead gaskets were placed above and underneath the collar. After placing heated tar in the filter, the cap, *P*, was screwed into place and compressed air was admitted through the connection, *L*. The operating pressure was usually kept close to 50 pounds per square inch (3.5 kg. per sq. cm.). The filtrate was collected in a 500-ml. Erlenmeyer flask immersed in an ice bath. In

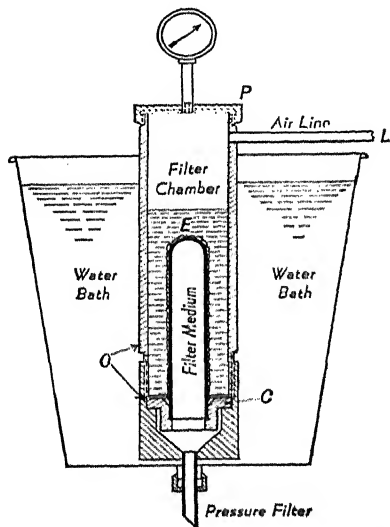


FIG. 326.—Apparatus for Removing Free-Carbon by Filtration.

spite of the relatively large filter area and the pressure employed, the operation of the filter was exceedingly slow. On the average, 150 to 200 ml. could be filtered before the cake had to be removed and the filter cleaned. The cycle required 6 to 8 hours.

After the end of the filtration, the cake was treated with two successive washings of 75 ml. each of nitrobenzene, and finally with 100 ml. of benzene. This also was done under pressure and the washings were rejected. The filter shell was then removed from the filter cylinder and placed in an air-tight container for vacuum evaporation. The evaporation was carried out over a period of at least 12 hours in a drying oven set to maintain a temperature

of 175° C. The average absolute pressure, maintained by a water ejector pump, was 10 mm. After this evaporation process the filter shell was weighed and the brittle cake removed by a spatula. The filter was then suspended in an electric furnace and the remaining carbon burned. The difference in weight between the loaded and the clean filter was used as a basis for the calculation of the amount of suspended phase in the tar. However, in most cases a correction had to be made for the volatile material still present in the cake. This correction factor was determined by placing a known quantity of the detached cake in a beaker and heating on a hot plate to 250–300° C. for 2–4 hours, after which there was no further visible evolution of vapors from the cake. The amount of volatile matter lost during this treatment was determined and used as correction factor. The amount of such vapor loss was usually less than 5 per cent of the weight of the cake.

Table CXXXIX shows the characteristics of representative tars before and after filtering.

(*V*) *Centrifugal Method*: The following rapid method has been proposed, involving the use of a centrifuge:²⁵⁵

About 10 g. of sample are weighed into a 250-cc. centrifuge bottle and heated to about 90° C. Twenty volumes of 7 per cent cresol-tetrahydronaphthalene solution (previously heated to about 90° C.) are added slowly, with manual stirring. The resulting mixture is stirred mechanically for approximately 3 minutes and then centrifuged at about 2400 rpm. for about 20 minutes. The supernatant layer is decanted and 150 to 200 cc. of benzene (room temperature) are added. The solid residue is broken with a stirring rod, after which the mixture is stirred mechanically for about 3 minutes and centrifuged. The layer of benzene is decanted, after which the bottle and residue are dried to constant weight at 110° C. (2 or 3 hours).

Test 24a. Solubility in Other Solvents. The following method has been described for ascertaining the insoluble matter in tars and pitches in sundry types of solvents, including acetone.²⁵⁶

The thin film of tar is produced on a circular section of fine-mesh wire gauze (2.5 cm. in diameter) to the center of which is attached a copper wire. This copper wire serves to suspend the

TABLE CXXXIX
SOLUBILITY OF REPRESENTATIVE COAL TARS

Type of Tar	Sam- ple No.	Condition of Tar	Float Test at 30° C., Sec.	Sp. Gr. at 25°/25° C.	Per Cent by Wt. Dist. up to 300° C.	Softening Point, ^a C.	No. Particles per Mg. of Tar	Quality of Suspended Phase	Suspended Phase in Per Cent by Wt. of Unfiltered Tar Determined from:				Per Cent by Wt. Insol. Exclusive of Suspended Phase in:			Temp. Coefficient of Viscosity	Surface Tension at 90° C., by Bubble Pressure, Dynes/Cm.
Vertical- retort	1-Y	Unfiltered	166.5	1.154	13.7	43.5	35,700,000	Coarse	Benzene insol.	Filtration	Microscope	(Sp. Insol.)	Benzene	Acetone	Ethyl ether	5.64	31.5
		Filtered	166.7	1.145	13.7	40.1			3.75	3.6	5.5	2.92	7.43	5.57	11.09		
	2-Y	Unfiltered	165.4	1.156	14.9	45.7	53,500,000	Coarse	2.55	3.4	3.9	3.55	7.56	5.25	11.43	5.05	31.4
		Filtered	165.7	1.150	16.8	44.4			2.55	3.4	3.9	3.55	7.56	5.25	11.43		
By-product coke-oven	3-C	Unfiltered	182.5	1.175	16.1	52.0	19,200,000	Very fine	0.44	1.00	0.5	1.00	3.32	7.54	12.17	6.53	35.7
		Filtered	182.5	1.172	17.1	52.0			0.44	1.00	0.5	1.00	3.32	7.54	12.17		
	4-C	Unfiltered	185.2	1.195	15.5	55.2	42,800,000	Very fine	0.65	1.62	1.4	1.62	5.47	7.46	15.75	6.55	37.5
		Filtered	185.2	1.193	16.4	53.3			0.65	1.62	1.4	1.62	5.47	7.46	15.75		
	5-C	Unfiltered	177.2	1.207	17.0	52.3	75,200,000	Very fine	2.76	1.91	4.0	3.6	6.12	10.56	21.1	6.49	39.8
		Filtered	177.2	1.202	17.0	52.2			2.76	1.91	4.0	3.6	6.12	10.56	21.1		
	6-C	Unfiltered	175.2	1.215	12.2	52.5	114,000,000	Fine	2.88	3.37	4.2	4.0	4.07	8.99	15.53	6.50	40.1
		Filtered	167.5	1.209	11.3	42.1			2.88	3.37	4.2	4.0	4.07	8.99	15.53		
	7-C	Unfiltered	185.5	1.218	13.6	51.4	94,200,000	Fine	5.47	4.71	5.7	5.6	6.47	11.44	25.2	6.35	36.8
		Filtered	185.2	1.207	17.3	52.7			5.47	4.71	5.7	5.6	6.47	11.44	25.2		
Horizontal- retort	8-H	Unfiltered	182.5	1.241	14.4	51.5	107,000,000	Very coarse	17.65	17.65	23.9	24.4	5.37	7.92	11.52	6.03	35.9
		Filtered	182.5	1.196	18.9	52.7			17.65	17.65	23.9	24.4	5.37	7.92	11.52		
	9-H	Unfiltered	176.5	1.270	18.9	67.5	281,000,000	Very coarse	21.67	21.32	23.9	23.2	6.63	11.33	22.5	5.76	35.6
		Filtered	176.5	1.202	22.9	52.4			21.67	21.32	23.9	23.2	6.63	11.33	22.5		

$$^b \frac{\log \eta_1 - \log \eta_2}{\log t_2 - \log t_1}$$

^a Ring and ball.

tar specimen in the solvent. Figure 327 illustrates the construction of this tar carrier.

After the carrier and the crucible through which the final solution is to be filtered have been weighed, a sample of the tar under investigation is heated in a beaker approximately 3 cm. in diameter to a temperature at which the tar is moderately liquid. The carrier is then lowered into the tar until the wire gauze is covered completely by it. It is then raised out of the tar, and the excess tar

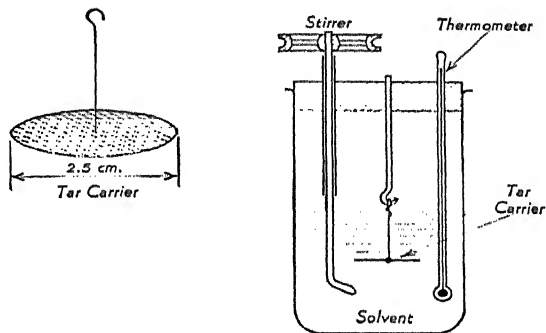


FIG. 327.—Apparatus for Solubility Determinations.

adhering to the gauze is removed by rapidly twirling the wire between thumb and forefinger. If this is done properly, the coating of tar left on the gauze is so thin that it is possible to distinguish the form of the individual wires of the wire gauze.

To determine the weight of tar adhering to the gauze, the carrier and Gooch crucible are weighed again. From the actual weight of the tar on the carrier, the amount of solvent to be used is calculated. The dimensions of the equipment used and a series of tests showed the ratio of 0.5 g. of tar to 200 ml. of solvent to be practical.

The amount of solvent required by this ratio is finally transferred into a tall 500-ml. beaker. The carrier is attached to a hook provided on the cork stopper of the beaker, and the stopper brought into position. An air-driven stirrer, also held in position by the cork stopper, is started and the digestion continued for 45 to 60 minutes.

Figure 327 gives a view of the assembled digestion apparatus. After digestion has been completed, the carrier is placed in the Gooch crucible and the solution is filtered. If small particles adhere to the walls of the tall beaker, the filtrate is transferred back into the digestion flask, agitated, and poured again through the filter. If necessary this procedure is repeated. Further purification of the solid residue in the crucible by washing with pure solvents is not deemed advisable, since they may have solvent powers different from those solvents in which some tar constituents have been dissolved. This holds true particularly in the case of solvents in which tars are soluble to only a relatively small extent.

Consequently, after completion of filtration, crucible and carrier were placed into a drying oven (110° C.) where they were kept

TABLE CXI.
RESULTS OF SOLUBILITY DETERMINATIONS

Type of Tar	Sample No.	Condition of Tar	Per Cent by Weight Insoluble in:				
			Carbon disulfide	Benzene	Acetone	Ethyl ether	Petroleum ether
Vertical-retort	1-V	Unfiltered	5.44	7.43	8.97	11.09	68.4
		Filtered	5.61	7.66	9.25	11.43	70.5
Coke-oven	2-V	Unfiltered	3.01	4.93	5.99	9.04	72.5
		Filtered	3.12	5.11	6.21	9.37	75.2
	3-C	Unfiltered	3.32	4.88	7.34	12.17	78.2
		Filtered	3.36	4.93	7.42	12.30	79.1
	4-C	Unfiltered	5.47	9.46	15.66	26.1	80.4
		Filtered	5.56	9.62	15.92	26.5	81.7
	5-C	Unfiltered	6.12	10.66	21.1	26.5	80.6
		Filtered	6.24	10.87	21.5	27.0	82.2
	6-C	Unfiltered	4.07	8.99	19.02	26.2	80.0
		Filtered	4.21	9.30	19.69	27.1	82.8
Horizontal-retort	7-C	Unfiltered	6.47	11.41	25.2	31.2	77.5
		Filtered	6.79	11.97	26.4	32.7	81.3
	8-H	Unfiltered	5.37	7.92	11.62	18.5	64.5
		Filtered	6.52	9.62	14.12	22.5	78.4
	9-H	Unfiltered	6.63	11.93	20.8	23.1	63.2
		Filtered	8.53	15.36	26.8	29.7	81.3

over a period of 5 to 10 minutes. When petroleum ether was used as a solvent, the drying period could not be extended over more than 5 minutes, since the residue still contained so much volatile material that prolonged heating caused evaporation of its more volatile portions.

Results obtained by this method are given in Table CXL.

(E) CHEMICAL TESTS

WATER

The estimation of water is made in some cases for purposes of identification, and in others as a criterion of the quality. Native asphalts and tars are examined in this way to ascertain whether

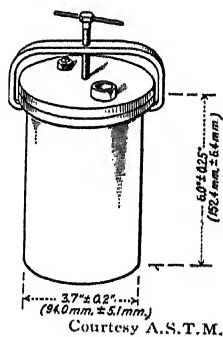


FIG. 328.—Copper Still.

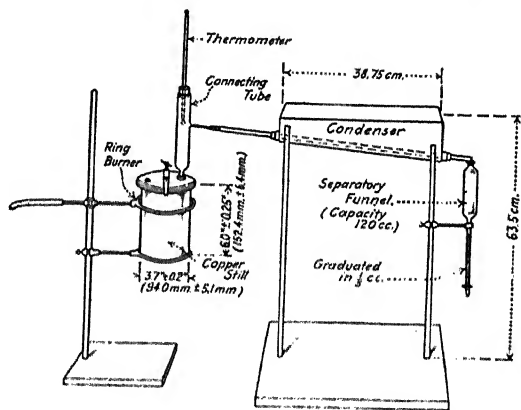


FIG. 329.—Assembled Apparatus for Water Test.

they exist in the crude or the dehydrated state. This test is also used for dehydrating bituminous substances to render them suitable for further examination, where the presence of water would interfere.

Test 25a. Substances Distilling at Low Temperatures.²⁵⁷ This method is adapted to crude petroleum, tars, creosote oil and other fluid bituminous substances distilling at comparatively low temperatures.²⁵⁸ A copper still provided with a removable flanged top and yoke of the form and approximate dimensions shown in Fig. 328.

An A.S.T.M. high-distillation thermometer, total immersion, graduated in either Centigrade or Fahrenheit degrees as specified, having a range of 0 to 400° C. or 30 to 760° F. A copper trough condenser, with straight-walled glass tube, having approximately the form and dimensions shown in Fig. 329. A separatory funnel

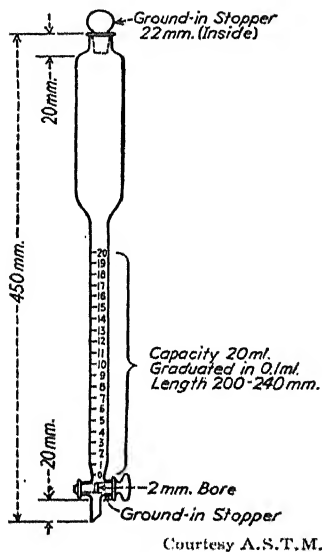


FIG. 330.—Glass Separatory Funnel, 200-ml. Capacity.

conforming to the requirements shown in Fig. 330. It shall have a total capacity of approximately 200 ml. with the lower 20 ml. graduated accurately in 0.1 ml. The graduation marks shall be numbered for each milliliter.

The apparatus shall be assembled as shown in Fig. 329. When any measurable amount of water is present in the distillate below 210° C. as determined in accordance with A.S.T.M. Designation: D 246, the oil and water in this fraction shall be separated, if possible, and measured separately. If more than 3 per cent of water is present, the percentage of water present shall be determined by the method described, and the water-free oil so obtained shall be used in the distillation test.

A 200-ml. sample of the oil shall be measured into a graduated cylinder and poured into a copper still, allowing the cylinder to drain into the still for several minutes. The lid shall be clamped on, using a paper gasket slightly wet with oil around the flange of the still. Heat shall be applied by means of the ring burner, which shall be placed just above the level of the oil in the still at the beginning of the test and gradually lowered when most of the water has distilled over. The distillation shall be continued until the vapor temperature reaches 205° C., as indicated by the thermometer with the bulb opposite the off-take of the connecting tube. The distillate shall be collected in a separatory funnel. When the distillation is completed, and a clear separation of water and oil in the funnel has taken

place, the water shall be read by volume and drawn off. Any light oil distilled over with the water shall be returned to the oil in the still. The dehydrated oil from the still shall be used for the distillation test.

To prevent frothing and spattering, it has been recommended²⁵⁹ that the still be surrounded with a cylindrical vessel, closed at the bottom and open at the top, of a somewhat greater diameter than the still. The vessel is so adjusted that its upper rim is a little lower than the level of the bituminous substance in the retort, whereupon it is filled with water until it overflows. Heat is then applied to the shallow layer of tar above the water level, with the ring burner. The water jacket absorbs heat which would otherwise be transmitted to the lower level of the tar and cause bumping. The level of water falls gradually by evaporation, permitting the zone undergoing dehydration to fall slowly. When the water level has fallen below the bottom of the still, the contents are practically dehydrated.

A modification of the foregoing method has been suggested²⁶⁰ which consists in providing the cover of the copper retort with two openings, one connected with a receptacle holding 100 ml. of tar and provided with a stop-cock to control the flow of tar into the retort. A weighed quantity of tar is introduced into the receptacle and enough run into the retort to cover the bottom, and the remainder is allowed to drip slowly into the retort. This prevents the tar frothing over.

Test 25b. Substances Distilling at High Temperatures. This method is adapted to asphalts and other bituminous substances (e.g., petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, etc.), comparatively free from volatile constituents, and incapable of distilling without suffering decomposition.

SUBSTANCES FUSING BELOW 300° F.

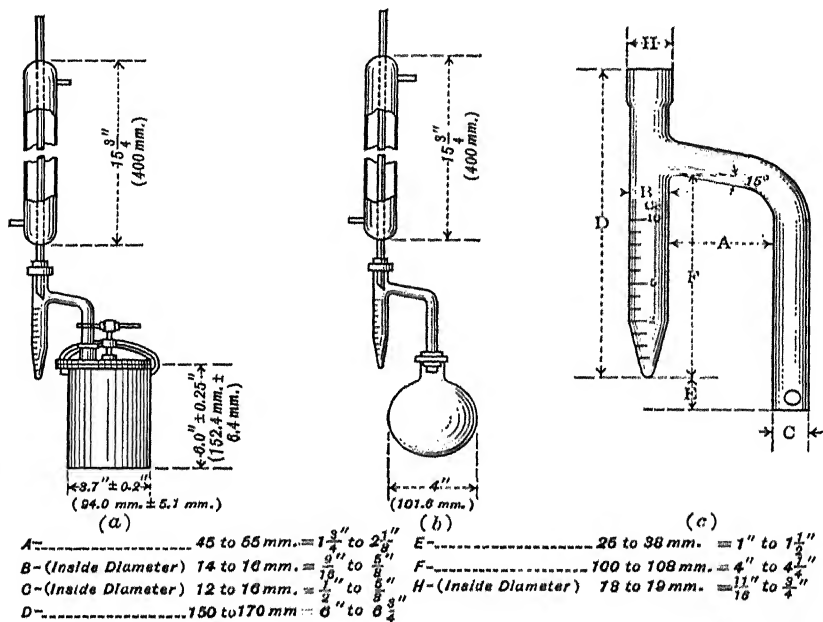
(1) *Exact Method:* When it is desired to determine the percentage of moisture without using the residue for other purposes, a convenient method consists in distilling it with a solvent.²⁶¹

The procedure has been standardized as follows:²⁶²

This method of test is intended for use in the determination of the water in a sample of bituminous material by distilling the sample with a volatile solvent. The method is suitable for a variety of

materials but is especially applicable to petroleum, fuel oil, road oil, coal tar, water-gas tar, coke-oven tar, and other petroleum products or bituminous materials.

The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging



Courtesy A.S.T.M.

FIG. 331.—Apparatus for Water Test.

into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method, but glass has been generally used for petroleum products and the metal still for road materials and tars.

(a) The metal still (Fig. 331a) shall be a vertical cylindrical vessel, preferably of copper, having a faced flange at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

(b) The glass flask (Fig. 331*b*) shall be of the short-neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

The burner used with the metal still shall be a ring gas-burner 4 in. (100 mm.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

(c) The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. (15¾ in.) in length with an inner tube 9.5 to 12.7 mm. (¾ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 deg. \pm 5 deg. from the vertical axis of the condenser.

(d) The trap shall be made of well-annealed glass constructed in accordance with Fig. 331*c* and shall be graduated from 0 to 10 ml. in 0.1 ml. divisions. The error of any indicated capacity shall not be greater than 0.05 ml. The outside diameters should be preferably 2.5 to 3.5 mm. (⅜ to ⅙ in.) greater than the inside diameters specified.

(e) The solvent used when testing petroleum products or bituminous materials derived from petroleum shall be gasoline, free from water, and shall conform to the following distillation requirements:

5 per cent shall distil at a temperature not below 194° F. (90° C.) nor above 212° F. (100° C.)

90 per cent shall distil below 410° F. (210° C.)

(f) The solvent used when testing bituminous materials derived from coal tar, water-gas tar, etc., shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements:

98 per cent shall distil between 248° F. (120° C.) and 482° F. (250° C.)

The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great so that the importance of sampling cannot be too strongly emphasized.

Procedure: When the sample to be tested contains less than 10 per cent of water, exactly 100 ml. of the material to be tested shall be placed into the still or flask and thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material. If the material is measured by volume, an accurate 100-ml. graduated cylinder shall be used and the contents transferred to the still by rinsing with one 50-ml. portion of solvent, followed by two successive 25-ml. portions of solvent, the cylinder being allowed to drain each time. When the sample to be tested contains more than 10 per cent of water, the volume of material used shall be decreased to that which will yield somewhat less than 10 ml. of water.

NOTE.—In special cases where the water content exceeds 10 per cent and it is not desirable to reduce the size of the sample to that which will yield somewhat less than 10 ml. of water, a distilling tube receiver graduated from 0 to 25 ml. may be used. This tube shall be graduated from 0 to 2 ml. in 0.1 ml., from 2 to 5 ml. in 0.2 ml. and from 5 to 25 ml. in 0.5 ml.

The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown. The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. below the surface of the liquid in the trap after stillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from two to five drops per second. The ring burner used with the metal still should be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds.

The distillation shall be continued at the specified rate until no more is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than an hour. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

or fused quartz combustion tube of 30 mm. internal diameter. The large heater *b*, 350 mm. long, surrounds the copper oxide, and the smaller one *a*, 200 mm. long, heats the sample in the boat. The combustion tube *d* of Jena glass or fused silica, measuring 21 mm. external diameter and 900 mm. long, is supported by an asbestos-lined nickel trough *e*. The current through each heater is regulated by separate rheostats *f* and *g*, the heating coils *a* and *b* requiring about 4.5 amperes at 220 volts.

The furnace is arranged so either air or oxygen may be passed through the combustion tube, and is equipped with two purifying trains in duplicate (of which but one is shown in the figure) connected to the combustion tube by a Y-tube, the joint being made tight by a rubber stopper. The purifying apparatus *II* contains the following reagents in order of the passage of the air or oxygen through them: sulfuric acid *i*, for removing any traces of ammonia; a 30 per cent potassium hydroxide solution *j*; granular soda-lime *k*; and granular calcium chloride *l*. One of the purifying trains is connected directly with an oxygen tank provided with a reducing valve for regulating the pressure, and the other being used for purifying the air supply, which is drawn through the apparatus by an aspirator connected with the other end of the combustion tube.

The first 3 cm. of the combustion tube are empty; then comes an asbestos plug (acid-washed and ignited); the next 40 cm. are filled with copper oxide gauze; then a second asbestos plug; then 10 cm. of fused lead chromate; and finally another asbestos plug 20 cm. from the end of the tube.

The absorption train consists of a 4-in. U-tube *m* filled with granular calcium chloride (previously saturated with carbon dioxide) to absorb the moisture. This in turn is connected to a Vanier

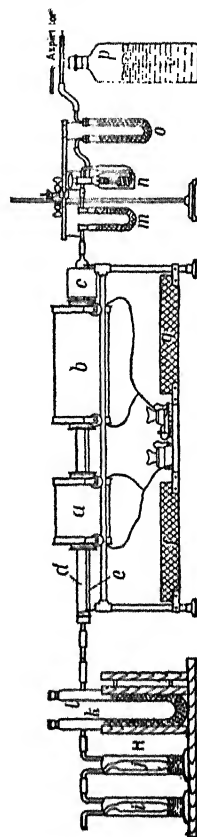


FIG. 333.—Combustion Furnace for Ultimate Analysis.

potash bulb *n* containing a 30 per cent potassium hydroxide solution and granular calcium chloride. The potash bulb is connected with an aspirator through the guard-tube *o* containing granular calcium chloride and soda-lime. A Mariotte flask *p* serves to keep the suction constant.

It is important to see that all connections are made tight. Before starting a determination or after any changes in chemicals or connections, a blank test should be run by aspirating 1 liter of air through the apparatus, which is heated in the same manner as though a determination were being made. If the Vanier bulb *n* or the calcium chloride tube *m* shows a change in weight of less than 0.5 mg. each, the apparatus may be considered in a satisfactory condition.

Approximately 0.25 g. of the bituminous substance is carefully weighed into a porcelain or platinum boat and transferred to the combustion tube which should be cool for the first 30 cm., the copper oxide at a bright-red heat, and the lead chromate at a dull-red heat. The boat should be introduced rapidly near the asbestos plug at the beginning of the copper oxide, the stopper connecting with the purifying train replaced and pure oxygen passed through at the rate of 3 bubbles per second. The current is *gradually* turned on heating coil *a*, which at the start should be at the right of the boat. By manipulating the rheostat, and gradually pushing the coil towards the boat, the evolution of volatile matter is carefully controlled to prevent too rapid an evolution of gas and tar, which may either escape complete combustion or be driven back into the purifying train. The heat should accordingly be increased slowly by manipulating the rheostat, until the sample ignites, whereupon the temperature may be increased rapidly. Any moisture collecting in the end of the combustion tube or in the rubber connection joining it to the calcium-chloride tube *m* is driven into the latter by carefully warming with a hot tile. After the sample ceases to glow, the oxygen is continued for 2 minutes, whereupon the heat is turned off, and 1200 ml. air aspirated through the train. The absorption bulbs are disconnected, wiped clean, allowed to cool and weighed. The percentage of carbon is equal to the increase in weight of the KOH bulb (*n*) multiplied by 27.27 and divided by the weight of the sample. The percentage of hydrogen is equal

to the increase in weight of the CaCl_2 tube (m) multiplied by 11.19 and divided by the weight of the sample.²⁶⁵

Test 28. Sulfur. This test is also used for differentiating between and identifying bituminous substances.

(1) *Quantitative Test:* A number of methods have been proposed for this purpose, but the most rapid and accurate one consists²⁶⁶ in igniting the substance in an oxygen bomb as follows:

The oxygen bomb shall have a capacity of not less than 300 ml., and shall be of a design or construction such that no leaks shall occur at any pressure or temperature generated during the test and such that when the bomb is open liquid contents can be easily and completely drained. The inner surfaces shall be of materials that are chemically and physically resistant to the process or products of combustion. The gaskets, insulating materials, etc., shall be, as far as possible, physically and chemically resistant and in no event shall they undergo any reaction which would increase or decrease the sulfur content of the bomb liquors.

The oil cup shall be of platinum, glazed silica, or other suitable material, with a capacity of not less than 2.5 ml. nor more than 5.0 ml. If a platinum oil cup is used, the fuse wire shall be of platinum; if a glazed silica oil cup is used, the fuse wire may be of either platinum or iron. No. 34 B. & S. gage is a convenient size.

(a) The distilled water and all reagents should be sulfur free, but in such cases where it is necessary to employ reagents not sulfur free, blanks shall be run and the figures thus obtained used to correct the results of actual determinations.

(b) **Barium Chloride:** The barium chloride solution shall contain 100 g. of $\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$ per liter.

About 5 milliliters of distilled water shall be placed in the bottom of the bomb. From 0.6 to 0.8 g. of the oil to be tested shall be placed in the weighed oil cup and the weight of this charge shall be determined to an accuracy of at least ± 0.002 g. The cup shall be placed in the proper position in the bomb, the ignition mechanism arranged and the bomb closed. Oxygen shall be admitted slowly until a pressure is reached as indicated by the following table:

Capacity of Bomb, ml.	Minimum Gage Pressure Atmospheres
300 to 350.....	40
350 to 400.....	35
400 to 450.....	30
450 to 500.....	27.5
Above 500.....	25

The leads from the firing circuit shall be attached, the bomb placed in a bucket of cold water, and ignited. The bomb shall be allowed to stand in the water for 10 minutes and shall then be removed. The valve of the bomb shall be opened, allowing the gas to escape at an approximately even rate, so that the pressure is reduced to atmospheric in not less than 1 minute. The bomb shall be opened, and all parts of its interior, including the oil cup, rinsed with a fine jet of distilled water. All washings, which should not amount to more than 350 ml., shall be collected in a beaker. Particular care should be taken not to lose, by splashing, or otherwise, any of the liquid contents of the bomb. The washings shall be filtered through a washed "qualitative" filter paper. The filter shall be washed thoroughly. Two milliliters of concentrated HCl and 10 ml. of saturated bromine water shall be added to the filtrate. The solution shall be evaporated to about 75 ml. on a steam bath or hot plate. Ten milliliters of hot barium chloride solution shall be added in a fine stream or drop-wise to the hot solution, stirring during the addition and for two minutes afterward. The solution shall be allowed to stand overnight, or shall be kept hot for one hour on the steam bath or hot plate, allowing the precipitate to settle for another hour, while cooling. The supernatant liquid shall be filtered through an "ashless quantitative" filter paper, the precipitate washed with water, first by decantation, then on the filter, till free from chloride. The paper and precipitate shall be transferred to a suitable weighed crucible, dried at low heat till moisture is evaporated, the paper charred (without flaming), and finally ignited at a good red heat till the precipitate is just burned white. A satisfactory means of accomplishing these operations is to place the crucible containing the wet filter paper in a cold electric muffle furnace and to turn on the current. Drying, charring, and ignition will usually occur at the desired rate. After ignition is complete,

the crucible shall be allowed to cool to room temperature, and weighed. The use of a desiccator is not recommended.

From the increase in weight of the crucible the percentage of sulfur shall be calculated from the formula:

$$\text{Percentage of sulfur} = \frac{\text{grams of BaSO}_4 \times 13.734}{\text{grams of substance used}}$$

The percentage of sulfur obtained by the same operator with the same apparatus should not differ by more than $0.02 + 0.04A$; while the percentages obtained by different operators in different laboratories should not differ by more than $0.02 + 0.08A$ (A represents the average of the percentage obtained).

A modified procedure consists in igniting 1 g. of the substance with 1 g. of an "accelerator" (potassium chlorate) and 0.2 g. of benzoic acid in a bomb calorimeter. The bituminous substance may be conveniently weighed on a small lump of chemically pure cotton (free from sulfur) and placed on a small platinum cone, which in turn is suspended from a copper wire. The cotton is connected with a thin platinum wire forming a short-circuit between the suspended copper wire and the return conductor.²⁶⁷

Another quantitative method, which is recommended as an alternative, where the bomb-calorimeter is not available, consists in weighing 1 g. of the substance in a 100 ml. porcelain or quartz crucible, adding 5 ml. concentrated nitric acid saturated with bromine, covering with a watch glass, digesting one-half hour on a steam bath, cooling, adding 1–2 g. anhydrous sodium carbonate small portions at a time, thoroughly mixing the contents, heating in an air-oven at 100°C ., and then igniting over a low flame, gradually increasing the heat until all the organic matter has been consumed. Cool, place in a beaker, add 200 ml. water, digest on a steam bath until dissolved, filter, wash, acidify with hydrochloric acid, using Congo red as indicator, adding 2 ml. acid in excess. Warm, add 10 ml. barium-chloride solution (100 g. per liter), digest on a steam bath and continue as described above.

The following method, involving the use of a catalyst has been proposed for the quantitative determination of sulfur in asphalts:²⁶⁸

Manganese chloride tetrahydrate (411 g.) is dissolved in 500

ml. of water; to this is added a solution of 255 g. of cupric chloride dihydrate in 400 ml. of water. The solutions are thoroughly mixed, warmed, and stirred. A solution of 500 g. of potassium hydroxide in 500 ml. of water is then added drop by drop, stirred with uniformity, washed by decantation several times, filtered on a Büchner funnel, and washed free of alkali and chlorides. The mixed oxides are then dried in the steam bath overnight and finally at 200 to 225°C. until all the moisture has been driven off.

About 0.25 g. of asphalt (divided into small pieces) is placed in a porcelain combustion boat (15 x 100 mm.) and enough chloro-

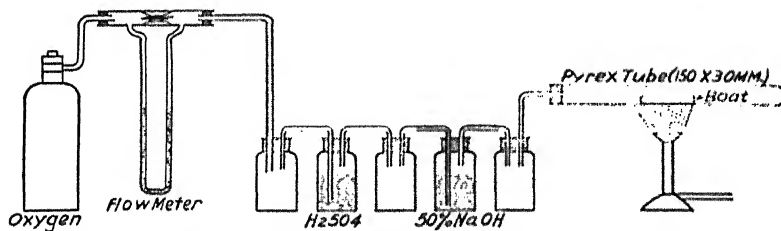


FIG. 334.—Determination of Sulfur by Combustion with a Catalyst.

form (about 3 ml.) is added to dissolve the sample. Enough of the catalytic combustion mixture is added to incorporate thoroughly with and cover the asphalt solution; this will usually require from 3 to 5 g. The chloroform is then allowed to evaporate, preferably by setting the boat on a steam bath. The combustion boat is inserted in an open Pyrex glass tube (150 x 30 mm. with 2-mm. wall), one end of which is fitted with a stopper carrying a tube leading to an oxygen generator as illustrated in Fig. 334. More than one boat may be used in a tube, provided it is sufficiently long and combustions are carried on concurrently.

The oxygen is preferably passed through a train comprising a 50 per cent sodium hydroxide solution, followed by a wash bottle containing concentrated sulfuric acid. Empty bottles are placed between these for safety.

The oxygen is passed through the tube in a fairly rapid stream, about 10 ml. per second, and the tube is heated, at the zone where the boat is located, with a fishtail burner with a low flame at the start. When the combustion starts, as evidenced by the glowing

of the asphalt-catalyst mixture, the flame is removed until the glowing ceases. To ensure complete oxidation, the tube is again heated for about one minute over a full flame. The oxygen is then shut off and the tube and boat are allowed to cool. When cool, the boat and contents are digested with boiling water, filtered through double ordinary filter paper, and washed with boiling water (containing 1 to 2 per cent of sodium carbonate in order to prevent turbid washings due to carrying through of colloidal matter). The filtrate is then made acid to Congo red with hydrochloric acid and refiltered on quantitative filter paper. A double filtration is desirable in order to free the filtrate completely from any colloidal matter that may have been carried through in the first filtration; quantitative paper is desirable. The sulfates are precipitated in the filtrate with barium chloride according to standard practice. Blank determinations for sulfur content of the catalytic combustion mixture should be made and corrections applied.

(II) *Qualitative Test*: A rapid test for detecting the presence of sulfur in cyclic bodies containing sulphur in ring formation (e.g., in asphalts, asphaltites, coal-tar pitch, etc.) consists ²⁰⁰ in separating the saponifiable constituents (which interfere with the reaction) from 10 g. of the substance by Test 39, or by dissolving in 25 ml. benzol with gentle heating, cooling and adding 30 ml. *N*/₂ alcoholic potash, shaking, and then rapidly diluting with 200 ml. 96 per cent alcohol. After standing a short time, the liquid (which should test alkaline to phenolphthalein) is decanted. The residue is washed with alcohol, dried on a water bath and finally at 105° C. It is then heated with 100 ml. of ether under a reflux condenser, and a few lumps of granular calcium chloride are added. After cooling, the liquid is filtered into a test tube to remove any insoluble matter present, and the solution mixed with 20 ml. 2 per cent mercuric bromide (HgBr_2) in ether, and allowed to stand overnight. If a precipitate forms, it is filtered off, washed with ether, and dissolved from the filter paper with warm benzol. If any sulfur-bearing bituminous substances are present, including petroleum or native asphalts, the precipitate will dissolve in the benzol forming a dark brown solution (any mercurous bromide present remaining undissolved). On evaporating the benzol, the mercuric-bromide-sulfur compound is deposited as a dark brown to black brittle mass.

A confirmatory test consists in heating the mercuric-bromide-sulfur compound with a few cubic centimeters fuming nitric acid on a water bath until it evaporates to dryness. The residue is dissolved in water and tested qualitatively for sulfur (by precipitating with barium chloride), for bromine (by precipitating with silver nitrate), and for mercury (by precipitating with ammonia and ammonium sulfide) respectively. Any sulfur compounds present in fatty-acid pitch (e.g., derived by heating with sulfur, or the like), unlike those present in natural or petroleum asphalts, are not precipitated by mercuric bromide, and this reaction may accordingly be used to advantage for detecting the presence of asphalt in fatty-acid pitches. It is claimed that 10 per cent of asphalt in fatty-acid pitch may be detected in this manner.

Test 29. Nitrogen. This determination is also used for identifying bituminous products, and the procedure ordinarily employed constitutes a modification of the well-known Kjeldahl-Gunning method.²⁷⁰

One to five g. of the bituminous material, which should be finely powdered when sufficiently hard, is boiled with 150 ml. of concentrated sulfuric acid, 50 g. of potassium sulfate, 2.5 g. mercuric oxide and 2.5 g. copper sulfate in a 500-cc. Kjeldahl flask for thirty minutes to two hours, depending upon how long it takes until the liquid attains a clear green color. The boiling should be continued at least two hours after the solution reaches the straw-colored stage, the total time required ranging from three to four hours. After the solution has cooled, a few crystals of potassium permanganate are added to insure complete oxidation. When thoroughly cool, the solution is diluted to 200 ml. with cold water, again cooled, and the following solutions added: 25 ml. of a 4 per cent solution of potassium sulfide to precipitate the mercury; 1-2 g. of granular zinc to prevent bumping; and finally enough saturated sodium hydroxide (usually 80-100 ml.) to make the solution distinctly alkaline, using phenolphthalein as indicator. The danger of losing ammonia may be minimized by holding the flask in an inclined position while the sodium hydroxide solution is being added and carefully running the alkaline solution down the side of the flask so it will form a layer below the acid solution. The flask should then be at once connected

with the condensing apparatus, and the solution mixed by gently rotating the flask.

The ammonia is then distilled into 10 ml. of standard sulfuric acid solution at the rate of 100 ml. per hour, until 150–200 ml. of distillate have passed over. The distillate is then titrated with standard ammonia or caustic soda solution, using cochineal as indicator with the former, or methyl orange with the latter (20 ml. standard ammonia or caustic soda solution are equal to 10 ml. of standard sulfuric acid, and also equivalent to 0.05 g. nitrogen).

A blank determination shall be made in exactly the same manner as described above, except that 1 g. of pure sucrose (cane sugar) shall be substituted in place of the sample. The nitrogen found in this blank determination shall be deducted from the result obtained with the sample.

Test 30. Oxygen (in Non-mineral Matter). There being no satisfactory direct method for determining oxygen, it is computed by subtracting the sum of the percentages of hydrogen, carbon, nitrogen, sulfur, water and ash from 100 per cent. The result so obtained is affected by all the errors incurred in the other determinations, and especially by the change in weight of the ash-forming constituents on ignition. Iron pyrites will absorb oxygen from the air and change to ferric oxide, increasing the weight of ash, and thereby causing a negative error in the oxygen, equivalent to three-eighths of the pyritic sulfur. Any calcium carbonate present will tend to absorb sulfur combined with the bituminous constituents. On the other hand, there is always a loss on ignition of "water of composition" from the clayey and shaley constituents, also carbon dioxide from carbonates, etc., which tend to compensate for the absorption of oxygen.²⁷¹

A more correct value is obtained by making the corrections indicated in the following formula:

Corrected Oxygen =

$$100 - [(C - C') + (H - H') + N + H_2O + S' + \text{Corrected Ash}]$$

Where C equals the total carbon; C' the carbon of carbonates; H the total hydrogen less hydrogen of water; H' hydrogen from water of composition in clay, shale, etc.; N the nitrogen; H₂O the

moisture as found at 105° C.; S' the sulfur not present as pyrite or sulfate; "Corrected Ash," the mineral constituents originally present, which for most purposes may be calculated with sufficient accuracy by adding to the ash as found, five-eighths of the weight of pyritic sulfur, the CO₂ of carbonates, and the water of composition of clay, shale, etc.

MOLECULAR WEIGHT

Test 30a. Freezing-point Method. This is determined by the usual Beckmann freezing-point method in a benzol or nitrobenzol solution, as described in any of the standard text books on physical chemistry. Sharp temperature readings can be obtained to 0.001°, and the weight of solvent should be recorded to within 0.01 g. This cryoscopic method is claimed to be accurate to ± 1.0 to 1.5 per cent.²⁷²

The following molecular weights have been reported:

Trinidad asphalt (soluble portion)	11,31.8
Bermudez asphalt (soluble portion)	6261.4
Gilsonite	4251.5

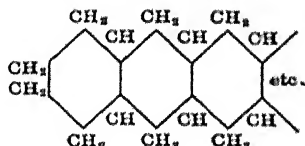
Asphaltenes have a molecular weight of about 2,400 and a carbon-hydrogen ratio of 11.1.²⁷³

Test 30b. Viscosity Method. The molecular weight (M) may also be ascertained from the viscosity (ν) at 120° C. (at which temperature the melted substance behaves as a true solution), by means of the following formula:

$$\log \nu = C.k.M.$$

where C = the concentration, and k = a constant. Tests indicate that asphalts have a molecular weight in the neighborhood of 1,800 at 120° C.²⁷⁴

Since asphalts have a polycyclic structure of the following typical formation, it may readily be calculated that Trinidad asphalt will consist of between 12 and 16 rings:



Test 30c. Vapor-pressure Method. The molecular weight may also be determined by the vapor pressure of a solution in toluol, or other solvent, compared with that of a standard solution of $(\text{:NPh})_2$ in the same solvent.

This variation of the vapor-pressure method is based on the principle that non-ionized and non-associated solutions of equal molecular concentration exert equal vapor pressures when enclosed in the same atmosphere, and by adjusting the concentration of one or the other, it is possible to arrive at a condition when there is no change in volume. Hence, if one solution is of known concentration, the molecular weight of the unknown is readily calculated. A suitable substance is C.P. azobenzene dissolved in commercial 90 per cent toluene to a concentration of $0.2/N$. A solution of the bituminous substance under examination is likewise prepared, by dissolving 2 g. in 100 ml. of toluene. Drops of these two solutions are alternately introduced into a capillary tube, in all 4 drops of the azobenzene and 3 drops of the bituminous solution. The ends of the capillary are then sealed and the tube immersed in water in a Petri dish. A number of such tubes are prepared, varying the concentration of the standard azobenzene solution (e.g., $0.2/N$, $0.1/N$, $0.05/N$, etc.). The lengths of the drops are measured under a microscope and the change in length noted after a time interval of say 12 hours. As an example, if the azobenzene solution was $0.1/N$ and the weight of the bituminous substance was 2.0 g. per 100 ml., then the molecular weight of the substance is at least 200. Some mean molecular weights as determined by this procedure are as follows: horizontal-retort coal-tar 240, low-temperature coal-tar 284, low-temperature coal-tar pitch 457, Mexphalte "E" 715, and gilsonite selects 1,510.^{27b}

TAR ACIDS

This test is used to ascertain the quantity of phenolic, cresylic, and other acid derivatives present in tars and pitches, especially those derived from non-asphaltic pyrobitumens, also from wood and bone. It is of value in establishing the quality for certain purposes, also as a means of identification. Two methods are employed for the purpose:

Test 31a. Contraction Method.²⁷⁶ One hundred ml. of tar or 100 g. of pitch are weighed into a tared Engler flask, and distilled by the flask method (Test 16b). With tars the distillation is continued until 95 per cent has been distilled off, and in the case of pitches it is stopped when the vapor temperature reaches 400° C. The time of distillation should occupy about twenty minutes, and the condenser tube heated to prevent the distillate from solidifying in it. The distillate is caught in a separatory funnel, the lower portion of which is graduated. This is immersed in water at 60° C. until no change in volume takes place, and a reading taken, whereupon 50 ml. of a 10 per cent caustic soda solution are added, shaken, allowed to settle, and the clear soda drawn off. The contents are brought again to 60° C., and the loss in volume noted. Shake with another 30 ml. of soda, and observe whether there is any further diminution in volume. If so, repeat until no further shrinkage occurs. The total shrinkage represents the *tar acids* present in the distillate.

NOTE.—The results obtained by this method are slightly higher than the true tar-acid content, for the reason that the soda withdraws certain compounds which are not subsequently liberated from the soda solution in the form of an oil, which substances are evidently acid in nature but not phenolic bodies.

Test 31b. Liberation Method. This method has been standardized as follows:²⁷⁷

This method of test covers the procedure for determining the amount of tar acids in fractions distilled from creosote and creosote coal-tar solutions.

(a) Type I Separatory Funnel: The type I glass separatory funnel shall conform to the requirements shown in Fig. 335. It shall have a total capacity of approximately 200 ml. with the lower 20 ml. graduated accurately in 0.1 ml. The graduation marks shall be numbered for each milliliter.

(b) Type II Separatory Funnel: The type II glass separatory funnel shall have a total capacity of approximately 260 ml. and shall conform to the requirements shown in Fig. 336. The capacity of the lower bulb from the stopcock to the first graduation mark shall be 65 ml., and above this mark the stem shall be graduated accurately for 100 ml. in 0.2 ml. The graduation marks shall be numbered for each 2 ml.

In making the determination, 100 g. of the sample shall be distilled in accordance with A.S.T.M. Designation: D 246. The fraction (Note) to be tested shall be transferred to a regular 250-ml.

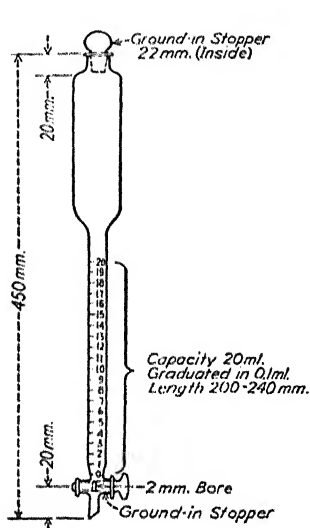


FIG. 335.—Type I Glass Separatory Funnel, 200-ml. Capacity.

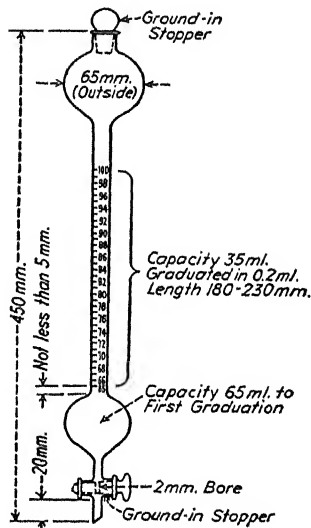


FIG. 336.—Type II Glass Separatory Funnel, 260-ml. Capacity.

Courtesy A.S.T.M.

NOTE.—If the total content of tar acids in a sample is desired, the entire distillate below 355° C. should be tested. In this case the results shall be reported as percentage by volume, that is, as the number of milliliters of tar acids per 100 ml. of the original dry sample, both volumes measured at the same temperature.

glass-stoppered separatory funnel, and 50 ml. of C.P. benzol and 50 ml. of an 18.3 per cent solution of NaOH (sp. gr. 1.20 at 20/4° C.) shall be added. The mixture shall be shaken vigorously for 3 min. and allowed to settle. The well-settled lower portion of the liquid shall then be drawn off into a 250-ml. beaker. An additional 30 ml. of the NaOH solution (18.3 per cent) shall be added to the separatory funnel and the contents gently shaken for 2 min. After settling, the lower portion of the liquid shall be drawn off and added to the beaker containing the first portion. Sufficient diluted H₂SO₄ (1 : 3) shall be added to the mixture in the beaker

to turn blue litmus paper definitely red. The solution shall be cooled during the addition of the acid.

If the tar acids liberated are estimated to be under 10 ml., type I separatory funnel shall be used and 10 ml. of high-flash naphtha or C.P. benzol at 25° C. shall be measured into it. The liberated tar acids and sulfate solution shall then be poured through this layer of naphtha or benzol several times, drawing the material off at the bottom of the funnel into the original beaker and pouring it back into the top of the funnel. This washes out the beaker and allows all the tar acids to be absorbed. The funnel shall then be allowed to stand at 25° C., until the layers separate clearly, when the sulfate solution shall be drawn off and the increase in the volume of the naphtha or benzol taken as the dry tar acids present.

If the tar acids liberated are estimated to be more than 10 ml., the procedure described shall be followed except that 65 ml. of high-flash naphtha or C.P. benzol at 25° C. shall be measured into the type II separatory funnel.

The result shall be reported as a percentage of the fraction tested, calculated as follows:

$$\text{Tar acids, per cent} = \frac{G \times V}{W} \times 100$$

where G = specific gravity of the tar acids at 25° C./15.5° C.,*

V = volume of tar acids in milliliters, and

W = weight of fraction in grams.

NAPHTHALENE

Test 32. Conventional Method. Naphthalene is present in tars and pitches generated at high temperatures, including those derived from coal. It is produced by the condensation of two or more hydro-carbon molecules, accompanied by the elimination of hydrogen. Tars used for impregnating felt should not contain more than 3 per cent naphthalene, as it will otherwise crystallize out on the surface of the sheet and gradually evaporate. Naphthalene is ascertained in the following manner. The oil which is

* For practical purposes the specific gravity of the tar acids may be assumed to be 1.040.

unacted upon after extraction of the tar-acids, as described in Test 31, is placed in a copper beaker and cooled with stirring to 40° F. and held at that temperature for fifteen minutes. The separated naphthalene shall then be quickly filtered on a paper in a perforated funnel, using suction, and the oil removed from the solids as quickly as possible. The solid cake shall then be removed from the filter with a spatula and pressed *repeatedly* in a letter-press between strips of filter paper until only a trace of oil is given up to the paper. The solids shall then be weighed and the percentage calculated.

The purity of naphthalene may be ascertained from its crystallizing-point, C.P. naphthalene showing a crystallizing-point of 80.2° F. Graphs have been devised showing the crystallizing-points of mixtures of pure naphthalene with varying percentages of creosote oil, phenols and heavy pyridine bases, respectively.²⁷⁸

SOLID PARAFFINS

Test 33. Solid Paraffins. Until recently, it was considered that the presence of paraffin was an indication of the quality of asphaltic products, and many specifications stipulated the maximum percentage permissible. It is now generally conceded, however, that there is no rational relation between the solid paraffins in asphaltic products and their quality. The determination of paraffin is therefore of value only for purposes of identification. Traces of solid paraffins are found in asphaltites, natural asphalts, and in asphalts produced from strictly asphalt-base petroleum. On the other hand, more or less paraffin is present in asphalts derived from non-asphaltic and semi-asphaltic petroleum. It is absent in tars and pitches derived from high-temperature distillation processes.

Various tests have been proposed from time to time for ascertaining the wax content of asphalts, involving: the destructive distillation of the asphalt;²⁷⁹ extraction of a mixture of the asphalt with fullers' earth;²⁸⁰ digestion with sulfuric acid;²⁸¹ etc. All such methods have been found to result in the alteration or partial destruction of the wax, with an adverse effect upon the accuracy of the results. Various solvents have likewise been suggested, includ-

ing: ether-alcohol and butanone (methyl-ethyl-ketone); ²⁸² isobutyl alcohol; ²⁸³ nitrobenzene; ²⁸⁴ propane; ²⁸⁵ methyl-n-butyl ketone; sec.-butyl acetate; trichloroethylene; a mixture of $C_2H_2Cl_2$ with methyl alcohol; ²⁸⁶ etc. The following procedures are claimed to give the most reliable results.

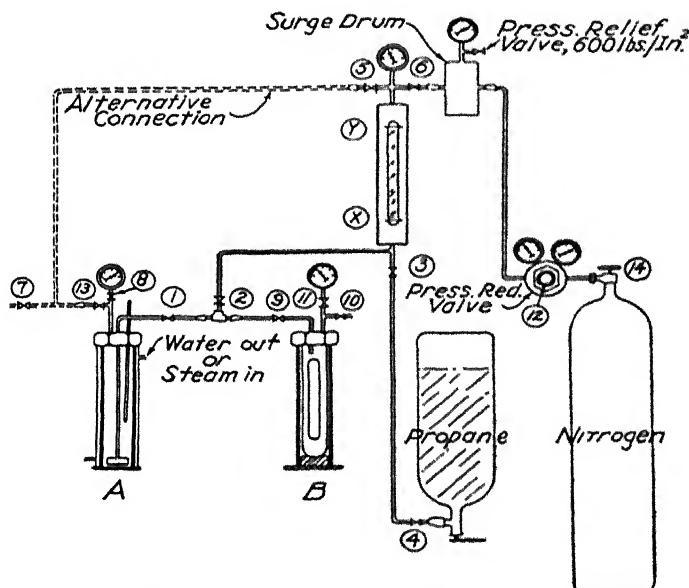


FIG. 337.—Assembly for Treating with Propane.

(I) *Propane-hexone Method.* This procedure consists in treating the asphalt with propane to separate the asphaltene-resin fraction from the oil-wax fraction, and subsequently isolating the wax from the latter by means of commercial "hexone" (methyl-isobutyl ketone) as follows: ²⁸⁷

The propane-treating equipment is shown in Fig. 337. This apparatus has been constructed so that it can be operated rapidly with complete safety. *A*, the precipitation vessel, is surrounded by a jacket through which hot water or steam is circulated to obtain the desired precipitation temperature of 68.3–71.1° C. (155–160° F.). The copper tube which extends nearly to the bottom of *A* has

a removable filter at the end which is made up of a 40-mesh copper screen, canvas cloth, filter paper, canvas cloth, and 40-mesh copper screen. Also attached at the bottom end of the filter tube and fitting inside the cylinder are two 40-mesh screen discs 1.27 cm. (0.5 in.) apart. The function of these discs is to collect the bulk of the precipitate and thereby prevent it from settling on the bottom of *A*, where it would tend to plug the filter. *B* is used as a transfer vessel and consists of a special high-pressure Pyrex glass tube fitted in a brass container with observation slots; it operates under a pressure of 123,000 kg. per sq. meter (175 lbs. per sq. in.) and serves to collect the propane-soluble matter (oil and wax).

The equipment for the dewaxing step is substantially the same as that employed by other investigators. Two methods of chilling have been used—namely, (1) a refrigerated brine-cooled bath which contained sheathed openings 44 by 140 mm., suitable for standard 120-ml. (4-ounce) bottles and a conical opening for the filtering funnel, and (2) a kerosine bath chilled with solid carbon dioxide. In either method the temperature was readily maintained at -17.8° or -26° C. (0° or -15° F.), as desired.

Weigh a 50-cc. beaker and stirring rod approximately, add 5.20 to 5.50 g. of asphalt sample, and record the gross weight of beaker, stirring rod, and sample to the second decimal. Add 11.0 ml. of benzene and weigh to nearest 0.1 g.; cover and warm on a steam bath until solution is complete. Reweigh, and if the heating procedure has reduced the amount of benzene to less than 8.8 g., add benzene to bring the weight of benzene to this figure, which is equivalent to 10 ml.

Then pour the benzene solution into *A*, avoiding splashing the walls. Place the beaker and stirring rod with adhering solution on the steam bath during the course of the remaining operations and finally weigh (± 0.01 g.) after the benzene has been completely evaporated.

The benzene solution of the sample having been transferred, screw the cover tightly into place and introduce 100 ml. of propane from the graduated propane reservoir, compressed nitrogen gas serving to assist in the transfer.

After closing the valves, detach it and thoroughly mix its contents by inverting and shaking for 30 seconds. Again attach to

the propane line, open valve 8, and place a thermometer in the well. Connect a steam line to the jacket inlet (top) and pass steam through until the temperature in the vessel reaches 68.3 to 71.1°C . (155 to 160°F .). The pressure in *A* will be at approximately $246,000$ kg. per sq. meter (350 lbs. per sq. in.) at this temperature. With these conditions maintained, allow the precipitated asphaltenes and resins to settle for 5 minutes or longer, after which transfer the propane solution of oil and wax, while still being heated, in the manner of a pressure decantation. Completion of the transfer will be indicated by the approximate volume, but primarily by the rapid drop of pressure. When the pressure drops to $211,000$ kg. per sq. meter (300 lbs. per sq. in.), $35,000$ kg. (50 lbs. per sq. in.) pressure of nitrogen is superimposed on the contents of *A* through the connections leading to valve 13. Wash the contents once with 100 ml. of propane and during this washing operation allow the contents to evaporate slowly. When the contents have settled, pressure-decant the propane washings from which the propane is permitted to escape as before. Remove test tube and wash contents into dish, using benzene as solvent, evaporate on steam bath, and record weight. This residue (oil plus wax) is then ready for the wax analysis.

Warm 2 to 3 g. of this residue on a steam bath to approximately 65.5°C . (150°F .). At the same time, warm 75 ml. of hexone to approximately the same temperature, then add enough of it to dissolve the sample. Transfer the dissolved sample to a 120 -ml. (4 -oz.) bottle and wash the dish into it with the remaining warm hexone. Cork and shake thoroughly to mix the sample. Warm the solution to 65.5°C . (150°F .) on a steam bath, then allow to cool to room temperature, before placing in an ice bath for 20 to 30 minutes. After precooling in this manner, bring the mixture to a temperature of -17.8°C . (0°F .) by immersing in a suitable bath; an hour in a bath a few degrees below -17°C . (0°F .) has been found satisfactory.

Suction-filter the precipitated wax through Whatman's No. 42 (12.5 -cm.) filter paper, wetted with cold hexone, on a funnel at -17.8°C . (0°F .). Shake the stoppered bottle containing the wax-solvent mixture gently to break the solid structure and carefully pour into the filter, avoiding suction to dryness before wash-

ing. When the bulk of the mixture has been transferred to the funnel, discontinue the suction before "cracks" are formed in the wax cake. Wash the sample bottle immediately and thoroughly with approximately 20 ml. of cold hexone at $-17.8^{\circ}\text{C}.$ ($0^{\circ}\text{F}.$) and transfer the washings to the filter with the bulk of the wax, saving the bottle with any adhering wax. Again apply suction and continue until the wax is free of solvent. Remove the paper and wax and dissolve this wax, as well as that which was left adhering to the bottle, through the paper with hot benzene. Evaporate the benzene solution on a steam bath, dry the residue at $105^{\circ}\text{C}.$ ($221^{\circ}\text{F}.$) for 30 minutes to remove traces of benzene and hexone, cool in a desiccator, weigh, and calculate the wax back in terms of the original asphalt sample.

(II) *Butyl-acetate and Aluminum Chloride Method.* This modification has likewise been found to give accurate results, and is performed in the following manner:²⁸⁸

A 20-g. sample of asphalt, weighed to the second decimal place, is spread over the walls of a 500-ml. balloon flask, heat being applied if necessary. The asphalt is gently refluxed with 200 ml. of $70^{\circ}\text{A. P. I.}$ naphtha until all tarry material has disappeared, then cooled slightly and mixed carefully with 5 g. of Filter-Cel (diatomaceous earth). The hot mixture is filtered with the aid of suction through a percolating tube 5 cm. (2 in.) in diameter and 35 cm. (14 in.) in length and containing a cotton plug covered with a 1.25-cm. (0.5-in.) layer of Filter-Cel. The flask and residue on the filter are washed with portions of hot naphtha (about $120^{\circ}\text{F}.$, equivalent to $48^{\circ}\text{C}.$), totaling 200 ml.

The solution of petroleues in naphtha is transferred to a 1000-ml. separatory funnel, hot naphtha being used to rinse the suction flask, and the separatory is placed in a steam-heated oven maintained at $125\text{--}130^{\circ}\text{F}.$ ($51\text{--}54^{\circ}\text{C}.$). When temperature equilibrium is reached, the solution is treated with 10 or 20 ml. of 98 per cent sulfuric acid, and the sludge is allowed to settle out. After the sludge has been withdrawn, the acid treatment is repeated until the volume of recovered acid is unchanged after treatment. The solution is then neutralized with 5°Bé. caustic soda and washed neutral to litmus, using a solution containing 25 g. of sodium sulfate

in a mixture of 300 ml. of alcohol and 700 ml. of water, the temperature being kept at 120 to 130° F. (49 to 54° C.).

When anhydrous aluminum chloride is used, the solution of petrolenes obtained as above is transferred to a 1,000-ml. balloon flask and refluxed for 0.5 hr. with 10 g. of the solid reagent. The mixture is then allowed to stand for 15 to 30 minutes at 120° F. (51° C.), when the supernatant solution is decanted to another flask, and the hot naphtha used to rinse the sludge is added thereto. The aluminum chloride treatment is repeated until there is only a slight formation of a red-colored sludge. Finally the solution is washed in a 1,000-ml. separatory at 120° F. (49° C.) with 50 per cent alcohol until the wash is neutral.

The neutral solution from either the acid or aluminum chloride treatment is dried in a 1- or 2-liter balloon flask by refluxing in a Dean and Stark apparatus until there is no further increase in the volume of water in the collecting tube, and then concentrated to 50 ml. by distillation. The concentrate together with small portions of hot naphtha used to rinse the flask is transferred to a tared 250-ml. beaker. The remaining naphtha is removed by careful evaporation on the steam bath and the resulting oily constituents are heated in an oven at 230° F. (110° C.) for 0.5 hr., then cooled and weighed to the second decimal place.

The dewaxing equipment used is similar to that generally employed, except that the funnel is made of brass rather than glass. This brass funnel, illustrated in Fig. 338, is of the Büchner type, and is fitted with a long barrel, *d*, threaded at one end, by means of which it can be attached to the funnel cone, *a*, in such a manner as to fit tightly against the perforated filter plate, *b*. To prepare the funnel the lower end of the filter tube is stoppered and sufficient dewaxing solvent added to fill the tube until the perforated filter plate placed in position *c* is just covered. A piece of snugly fitting No. 10 duck filter cloth, *e*, is then placed on the plate and the funnel barrel is inserted and screwed down tightly, thus firmly clamping the filter cloth in position. A 0.6-cm. (0.25-in.) layer of Filter-Cel is placed on the cloth and moistened with 10 ml. of *sec*-butyl acetate, and the assembled tube is placed in a bell-shaped funnel which serves as a cooling bath.

A 3 ± 0.01-g. sample of the oily constituents is dissolved in 25

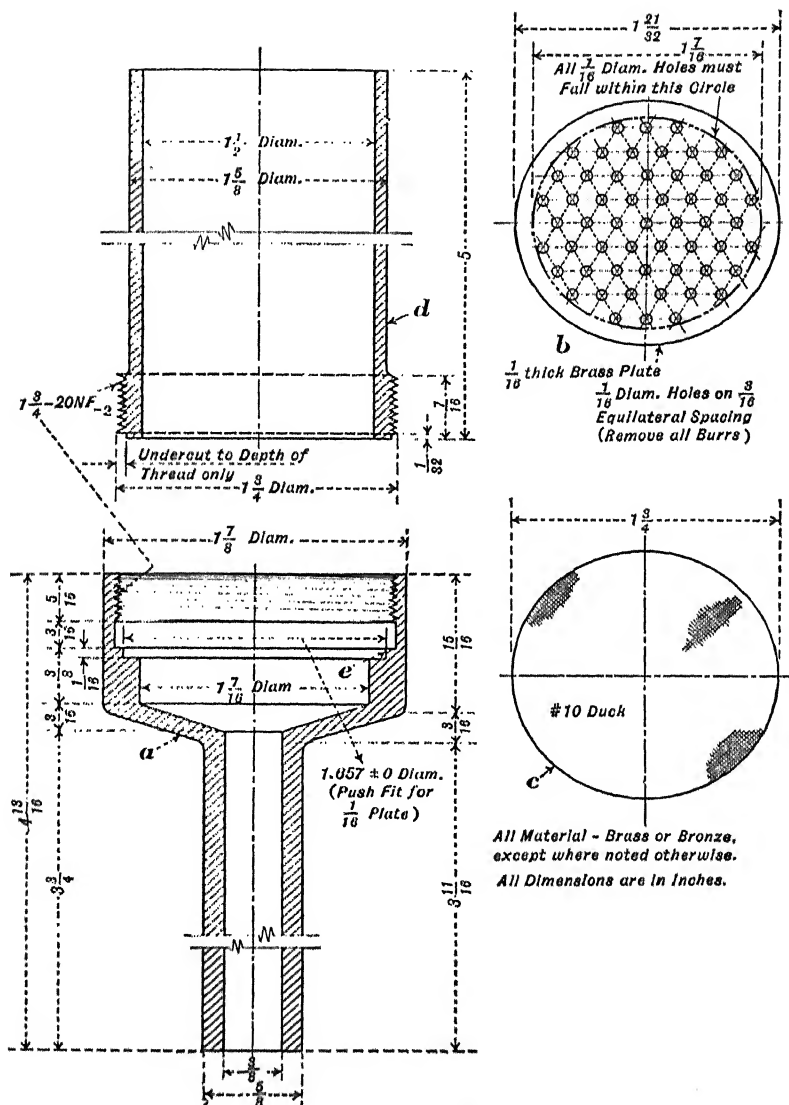


FIG. 338.—Wax Filter.

ml. of *sec*-butyl acetate in a 50-ml. beaker, and warmed on a hot plate sufficiently to obtain a clear solution. The resulting solution is transferred to the prepared filter tube, using 25 ml. of warm *sec*-butyl acetate to rinse the beaker. After the contents of the tube have been gently stirred, the tube is stoppered with a cork through which passes a thermometer adjusted so that its bulb is immersed in the solution. Alcohol is now added to the bell-shaped funnel until it is within an inch of the funnel top, and three or four stoppered test tubes, each containing 20 ml. of *sec*-butyl acetate, are immersed therein. The bath is chilled by the addition of dry ice and, during chilling, the contents of the tube are stirred occasionally until a temperature of -4° F. (-20° C.) is reached, which is maintained constant for 15 minutes. The cork is removed from the bottom of the filter tube, filtration is then carried out with the aid of suction, and the wax cake is stirred vigorously with a portion of prechilled solvent and again filtered.

Washing is repeated until the issuing liquid is colorless, then the cold alcohol is removed from the bath, and the apparatus allowed to come to room temperature. The filter assembly is removed, dried with a towel, and attached to a 250-ml. suction flask. Hot 70° A. P. I. naphtha is added to the tube, stirred vigorously, and withdrawn with suction. Then naphtha (20 ml.) is added to the tube and, after the side arm of the suction flask has been stoppered, the combination is placed on a hot plate and refluxed until condensate appears at the funnel top. After cooling slightly, filtration is carried out with suction and the tube rinsed with 70° A. P. I. naphtha. The filtrate thus obtained is evaporated in a tared crystallizing dish on a steam bath and the resulting wax is heated at 230° F. (110° C.) to constant weight, ± 0.001 g.

Wax melting-points are obtained by the rotating thermometer method. A small quantity of wax is placed on the bulb of a thermometer and warmed until the wax is in a molten condition, the thermometer is inserted in a test tube to protect it from air currents, then gently rotated, and the temperature at which solidification occurs is recorded as the melting point.

The *refractive index* of the solid paraffins will indicate whether the original substance was ozokerite or paraffin, or a mixture of the two. When tested at 90° C. on the Zeiss or Abbe refractom-

eter, ozokerite will show a refractive index below 15.0, whereas the solid paraffins derived from petroleum, shale, lignite, etc., will test between 15.0 and 30.0.²⁰⁰ When tested in the solid state, commercial paraffin waxes show a refractive index ranging from 1.500 to 1.540 at temperatures between 30 and 130° F.²⁰⁰ Furthermore, paraffin wax has a lower molecular weight than ceresine or ozokerite—for melting points between 50 and 60° C., the former tests between 330 to 400, and the latter above 450.

The penetration and melting-point relationship of the principal commercial waxes are as follows:²⁰¹

	Melting-point	Penetration at 77° F.
Montan wax.	74° C.	5
Carnauba wax.	83° C.	1
Ozokerite-ceresine.	92° C.	5
Borneo paraffin wax.	63° C.	11
Synthetic wax.	96° C.	32 *

* Much softer than pure waxes.

A method has also been proposed for detecting the presence of montan wax in admixture with asphalts,²⁰² and a procedure has been standardized for ascertaining the oil content of commercial paraffin wax.²⁰³

SULFONATION RESIDUE

Test 34 . Residue Insoluble in Concentrated Sulfuric Acid.

This method of test has been proposed for separating saturated and unsaturated hydrocarbons, as a means of distinguishing various asphaltic products (e.g., native asphalts and petroleum asphalts) from tars and pitches, thereby serving for purposes of identification. Two alternate procedures are available, viz.:

(1) *Portion Soluble in 88° Petroleum Naphtha.*²⁰⁴ The portion soluble in 88° petroleum naphtha, separated as in Test 23, is brought to exactly 100 ml., either by adding more 88° naphtha or else by evaporation, so that the quantity of substance carried in solution will be approximately 5 grams. This is then shaken in a 500 ml. separatory funnel at 77° F. for *exactly* three minutes, with 30 ml. of a mixture of concentrated sulfuric acid and fuming sulfuric acid, having a specific gravity of 1.84 at 77° F. The funnel is allowed to stand quietly overnight, whereupon the acid is drawn off and the oils unacted upon treated with another 30 ml. of the acid. This time a few hours' standing should effect a sharp sep-

aration. If the second acid layer is strongly colored, the treatment should be repeated a third time. The naphtha solution is washed successively with water, a 5 per cent solution of sodium carbonate and finally with water. The solution is evaporated to dryness over a steam bath and the residue weighed. This is equal to the saturated hydrocarbons present in the portion soluble in 88° petroleum naphtha. As a guide in evaporating the last traces of naphtha from the saturated hydrocarbons, a blank test should be run on 100 ml. of the 88° naphtha, whereupon the portion unacted upon is mixed with 0.75 g. of a non-asphaltic petroleum residuum and evaporated on the steam bath alongside of the sample under test, until the former is reduced to exactly its original weight.

The results are expressed as the percentage of saturated hydrocarbons present in the portion soluble in carbon disulfide (Test 21). This is calculated in the following manner: If a represents the percentage soluble in carbon disulfide, b the percentage soluble in 88° naphtha and c the percentage of saturated hydrocarbons in b ; then the saturated hydrocarbons present in the portion soluble in carbon disulfide will equal $\frac{bc}{a} \times 100$.

(II) *Distillate at 300–355° C.* This test expresses the percentage of saturated hydrocarbons in the distillate between 300 and 355° C. obtained upon subjecting the bituminous substance to the flask method of distillation (Test 16*b*). It is used to differentiate tars and pitches among themselves as well as from mineral waxes, asphalts (native and pyrogenous) and asphaltites. The figures for coal-tar pitches have already been given. The author cites the following additional figures: wood-tar pitch 0 per cent, saponifiable fatty-acid pitches 0 per cent, unsaponifiable fatty-acid pitches 0 per cent, residual asphalt from Mexican asphaltic petroleum 86 per cent, wurtzilite asphalt 87 per cent and gilsonite 85 per cent.

The following procedure has been standardized:²⁰⁶

This method of test is intended for use in determining the amount of unsulfonated residue in the total distillate to 300° C., or in the fraction of the distillate from 300° C. to 355° C. obtained by means of the Standard Method of Test for Distillation of Tars and Tar Products.

The following reagents are required:

(a) Sulfuric Acid (37 *N*): Prepare 37 *N* H_2SO_4 by blending reagent-grade fuming and concentrated sulfuric acids to 98.61 ± 0.2 per cent H_2SO_4 , as determined by titration.

(b) Sulfuric Acid (*Sp. Gr.* 1.84).

The following apparatus is required:

(a) **Test Bottles:** The test bottles shall be made of good quality glass and shall be 6 in., 18 g., either 8 or 10 per cent Babcock milk-test bottles. The capacity to the base of the neck shall be 45 to 50 ml. The graduated portion of the bottle shall contain 1.60 ± 0.025 ml. for the 8 per cent test bottle and 2.00 ± 0.025 ml. for the 10 per cent test bottle at a temperature of 77° F. (25° C.). The 8 per cent test bottle shall be graduated in 80 divisions and the 10 per cent test bottle shall be graduated in 100 divisions, the first and each succeeding tenth line to extend at least three-fourths of the distance around the neck and to be numbered from the bottom 1, 2, etc.

Within the range from 0–8 for the 8 per cent test bottle and 0–10 for the 10 per cent test bottle, the maximum error in volume shall not be greater than 0.025 ml. The graduation marks shall be clear and fine, not more than 0.3 mm. in width. The bottom of the bottles shall have a ground area of at least 2 sq. cm. for numbering.

(b) **Water Baths:** Two water baths, as follows: A water bath maintained at $77 \pm 0.5^{\circ}$ F. ($25 \pm 0.3^{\circ}$ C.) and of such depth that the contents of the test bottle are immersed below the surface of the water for the final reading.

A water bath maintained at 208 to 212° F. (98 to 100° C.) and of sufficient depth to permit complete immersion of the body of the test bottle.

(c) **Centrifuge:** A centrifuge capable of whirling two or more test bottles filled with acid at a speed of 1,000 rpm. The centrifuge shall be of good design and rugged construction so that it may be operated without danger.

(d) **Burette:** One 50-ml. burette graduated in 0.1-ml. divisions.

Procedure: 10 ± 0.1 g. of the distillate, or fraction of distillate, shall be weighed into the test bottle. If the distillate contains solid matter, the distillate shall be warmed in a hot water bath, with stirring, until the solid matter has melted before the sample for testing is taken.

Then 10 ml. of 37 *N* H_2SO_4 shall be slowly added to the test bottle from the burette in such a way as to wash down any oil remaining in the neck of the bottle. The test bottle shall then be shaken vigorously for 2 min. The temperature of this acid-distillate mixture shall not be allowed to approach 212° F. (100° C.) as indicated by the bottle becoming too warm to touch, or by the

contents foaming excessively. The test bottle may be cooled in ice water if necessary. However, if the distillate contains solid matter that does not readily disperse in the acid, it may be necessary to warm the distillate-acid mixture in the hot water bath to liquefy the solid matter.

An additional 10 ml. of 37 *N* H_2SO_4 shall be added as outlined and the bottle shaken vigorously for 30 sec. The test bottle shall then be placed in the water bath at 208 to 212° F. (98 to 100° C.).

After the test bottle is in the bath for 10 min., it shall be removed, shaken vigorously for 30 sec., and replaced immediately in the water bath at 208 to 212° F. (98 to 100° C.). If the sample boils or foams over at this stage, it shall be discarded and the test repeated.

The procedure outlined shall be repeated for a total of six immersions and shakings. However, 10 ml. of 37 *N* H_2SO_4 shall be added after the third immersion. After the last shaking the bottle shall be allowed to cool.

After cooling the test bottle to approximately room temperature, sufficient H_2SO_4 (sp. gr. 1.84) shall be added to the contents of the test bottle to raise the liquid level in the neck to the top of the graduation. The test bottle and its contents shall then be placed in the centrifuge and whirled at a speed of approximately 1,000 rpm. for 5 min. The bottle shall then be removed from the centrifuge and placed in the water bath at 77° F. (25° C.) so that the contents of the bottle are immersed below the surface of the water. After 10 min., the test bottle shall be removed from the bath and the volume of oil read to within 0.02 ml., which is one-tenth of a division. The centrifuging shall be repeated until the volume of oil is constant.

After a constant oil volume has been obtained by centrifuging, the reading of the bottom of the oil level shall be subtracted from the reading of the top and the difference, in terms of the major divisions, multiplied by 2. This value so obtained is the milliliters of unsulfonated residue per 100 grams of distillate. The sulfonation index is obtained by multiplying the milliliters of unsulfonated residue per 100 g. of distillate by the percentage by weight of the distillate of the tar, and dividing by 100. The sulfonation index shall be reported to the nearest 0.1. Due attention shall be paid

to calibration corrections. If the unsulfonated residue is dark in color, it shall be treated with an excess of 10 per cent sodium hydroxide solution, and if completely soluble in this reagent, the test is regarded as negative.

In case the volume of the unsulfonated residue exceeds the capacity of the graduated stem, the test shall be repeated using 5 g. of distillate instead of 10 g. of distillate, and the reading shall be multiplied by 4 to obtain the milliliters of unsulfonated residue per 100 grams of distillate. The sulfonation index is then computed.

Precautions: (a) It is extremely important that all glassware used in this test shall have been thoroughly cleaned and dried before use.

(b) If at any time during the test even the smallest quantity of oil or acid-oil mixture is lost, the sample shall be discarded and the test repeated.

(c) The rate of whirling may be decreased to avoid breakage of the test bottles. In all cases, however, the centrifuging shall be continued until a constant reading of the volume of unsulfonated residue is obtained.

Test 34b. Residue Insoluble in Water.²⁰⁶ Treat 3 g. of the carbon disulfide or benzol soluble portion of the substance, after evaporation of the solvent, with 6 ml. of concentrated sulfuric acid (95 per cent) in a stout test-tube and agitate continuously for three-quarters of an hour while heating in boiling water. The contents of the test-tube are then poured into 500 ml. of cold water. Brown-coal-tar and brown-coal-tar pitch are practically insoluble in cold water, and partly soluble in hot water. Coal-tar pitch, wood-tar pitch, oil-gas-tar pitch and naphthol pitch are completely soluble in cold water, forming a deep black solution, which upon filtering leave little to no residue. Lignite-tar pitch, peat-tar pitch, bone-tar pitch, rosin pitch, fatty-acid pitch, wool-fat pitch and montan pitch are partly soluble in cold water. Asphalts (both natural, as well as those derived from petroleum) and asphaltites are insoluble in cold water, forming a copious insoluble precipitate.²⁰⁷ Let the solution stand for two hours and without agitating the precipitate, decant carefully through a weighed Gooch crucible of about 7 cm. diameter. Wash the residue with hot water, and finally transfer the precipitate to the filter. Continue washing with water until the

filtrate no longer gives an acid reaction with methyl orange, then dry at 105° C. and weigh. Marcusson used this method for determining the percentage of coal-tar pitch in admixture with refined Trinidad asphalt. He found that upon treating the CS₂ soluble portion of this asphalt with sulfuric acid as outlined above, a small amount of soluble sulfo-derivatives was formed, so that an average correction of 4 per cent must be made to obtain accurate figures, as shown in Table CXLII.

TABLE CXLII

SEPARATION OF MIXTURES OF TRINIDAD ASPHALT AND COAL-TAR PITCH

Crude Trinidad Asphalt (Containing 45% Soluble Constituents), Per Cent	Refined Trinidad Asphalt (Containing 58% Soluble Constituents), Per Cent	Coal-tar Pitch, Per Cent	CS ₂ Soluble Constituents of the Asphalt Present in the Mixture			
			Per Cent Calculated	Per Cent Found	Per Cent Corrected (+4.0%)	Per Cent Difference
15	85	6.8	4.2	8.2	+1.4
20	80	9.0	6.4	10.4	+1.4
25	75	11.3	8.4	12.4	+1.1
30	70	13.5	11.0	15.0	+1.5
40	60	18.0	13.4	17.4	+0.6
45	55	20.3	14.6	18.6	+1.7
60	40	27.0	22.8	26.8	+0.2
.....	20	80	11.6	7.3	11.3	+0.3
.....	23	77	13.4	9.2	13.2	+0.2
.....	30	70	17.4	12.3	16.3	+1.1
.....	35	65	20.3	14.9	18.9	+1.4
.....	40	60	23.2	16.9	20.9	+2.3

The foregoing test has been modified for the ostensible purpose of yielding more accurate results, as follows: ²⁰⁰ 2 g. of the substance are weighed in a test-tube 3.5 cm. in diameter, which is immersed 12 cm. in a 30 per cent solution of salt, maintained at 107–108° C., whereupon 20 g. of concentrated sulfuric acid (95 per cent) are added. A mechanical glass stirrer is introduced and caused to revolve at the rate of 300 turns per minute and the digestion continued for 2½ hours. The mechanical stirring is claimed to break up the lumps and result in a better filtration. Carefully introduce water in the test-tube, to a point 12 cm. from the bottom and continue the stirring for another 15 minutes, then

transfer the contents of the tube into 1 liter of boiling water. Let stand overnight and carefully filter through two layers of tared filter paper in a 9 cm. porcelain filter, using suction. Wash the residue with boiling water until the filtrate is clear and no longer reacts acid with methyl orange. The filter containing the residue is placed on a Petri dish and dried for 2 hours at 105° C., cooled in a desiccator and weighed. This represents the asphaltic constituents present, which, however, are contaminated with a certain proportion of *insoluble* sulfo-derivatives of the asphalt. An examination of a range of petroleum asphalts, as well as Trinidad asphalt, indicated that the weight of insoluble asphaltic constituents, with their associated insoluble sulfo-derivatives, as ascertained by this method must be corrected by deducting 20 per cent, to obtain the net weight of asphaltic constituents actually present. Similarly, an examination of a number of coal-tar pitches showed that in no case was more than 0.1 per cent of insoluble sulfo-derivatives present. No correction need therefore be made for pitches. Any free carbon present in the pitch (ascertained by the insoluble matter separated upon originally dissolving the substance in carbon disulfide) should be taken into account by using the following formula:

$$\text{Per cent asphalt} = 80\% (100 - C) \cdot \frac{S}{E}$$

where C = per cent free carbon present in the mixture,

S = grams residue obtained after sulfonation,

E = gram mixture (after having been freed from insoluble constituents and carbonaceous matter) taken for the sulfonation process.

Heinrich Mallison claims²⁰⁰ that asphalts are converted into both soluble and insoluble sulfo-derivatives, and that certain asphalts when sulfonated by themselves showed slight increase in weight, whereas when sulfonated in admixture with 85 per cent of coal tar, they were converted almost quantitatively into soluble products.

It has been observed^{200a} that when dry HCl gas is passed through a solution of asphalt in carbon disulfide, a copious pre-

precipitate will form in the case of gilsonite, whereas none to a trace is obtained with other types of asphalt. This constitutes a simple method for identifying gilsonite.

Test 34c. Dimethyl Sulfate Method. This was originally proposed as a qualitative method for detecting mixtures of pitches with asphalts. It is based upon the fact unsaturated hydrocarbons are soluble in dimethyl sulfate, whereas saturated hydrocarbons are not. The material is distilled as in the preceding test, and 4 ml. of the distillate shaken with 6 ml. of dimethyl sulfate in a 10 ml. cylinder graduated to 0.1 ml. After standing, the percentage of insoluble hydrocarbons (i.e., saturated) is read off.³⁰⁰ The results are only approximate. Diethyl sulfate has also been proposed for the purpose, and is claimed to be more reliable.³⁰¹

FORMOLITE REACTION

Test 35. Nastjukoff Method. This is based upon the observed fact that cyclic unsaturated hydrocarbons, resins and asphaltic constituents yield insoluble formolite, whereas paraffins, olefins, naphthenes, polynaphthenes, ketones and sulfur compounds remain unchanged.³⁰²

The test is performed as follows: Dissolve $\frac{1}{2}$ to 1 g. of the substance in 35 ml. carbon tetrachloride, cool in an ice-bath and add 15 ml. concentrated sulfuric acid, drop by drop, with constant agitation. While still on the ice-bath, add 5 ml. of 40 per cent formaldehyde, drop by drop, and allow the mixture to stand for fifteen minutes. Dilute upon cooling with 100 ml. water and neutralize with strong ammonia. After standing fifteen minutes, decant the liquor through a tared Gooch crucible. Wash the residue in the flask with water and then with CCl_4 , and pour the washings through the filter. The contents of the flask are then washed into the filter with water and CCl_4 , and washed alternately with water and CCl_4 until the washings run through clear. The residue is then dried in an air-bath at 220°F. for one hour, cooled and weighed. The weight of the residue is calculated in percentage of the original sample, and represents the "formolite residue." It has been found that upon blowing asphalts, there is an increase in the amount of formolite residue.³⁰³

DEGREE OF MERCURATION

Test 36. Conventional Method. This is based upon the fact that mercuric acetate reacts with unsaturated hydrocarbons and sulphur compounds, accompanied by the liberation of acetic acid.³⁰⁴ A benzol solution of the bituminous substance is shaken with a solution of $\text{Hg}(\text{OAc})_2$ in methyl alcohol, and the liberated acetic acid is estimated by titration. The number of milliliters of N/KOH required to neutralize the acid liberated by 100 g. of the substance is termed the "degree of mercuration." A solution of mercuric nitrate (Millon's reagent) has been proposed,³⁰⁵ to detect coal-tar products in admixture with asphalt. This is accomplished by boiling 10 g. of the substance with 25 ml. N/NaOH for 20 minutes, filtering and adding nitric acid until almost neutral. Then concentrate to 5–10 ml., add 5 ml. of Millon's reagent and heat the mixture in a test-tube in boiling water for 30 minutes. The presence of even 1 to 2 per cent of coal tar or coal-tar pitch will cause a coloration, whereas no color will develop with asphalt or rosin.

SAPONIFIABLE CONSTITUENTS

In the case of bituminous materials, the estimation of the unsaponifiable and saponifiable matters is of value for purposes of identification. Certain bituminous substances, such as montan wax, rosin pitch, and fatty-acid pitch, are often composed largely of saponifiable constituents. Others, including pine-tar, pine-tar pitch, hardwood tar, hardwood-tar pitch, peat tar, lignite tar, bone tar, bone-tar pitch and other forms of fatty-acid pitches, contain smaller percentages. This test is also used for gauging the uniformity of supply, and in the case of fatty-acid pitches, as a criterion of the quality.

The relation between the acid, lactone, ester and saponification values, also the unsaponifiable and saponifiable constituents, is shown in Table CXI.III.

Test 37a. Free Acids ("Acid Value"). Boil 5.00 g. of the material under a reflex condenser with 25 ml. of benzol until the material dissolves, or else is completely disintegrated, then add 100 ml. of carefully neutralized 95 per cent ethyl alcohol and con-

tinue boiling for twenty minutes.* The liquid is decanted from the insoluble residue while hot, the latter boiled with another 50 ml. of alcohol, and the process repeated, until the extract no longer reacts acid with alkali blue 6-B (or phenolphthalein). The residue is then disregarded. To the combined extracts, add 10 ml. of a 25 per cent barium chloride solution and 6 drops of a 3 per cent alcoholic solution of alkali blue 6-B (or an equivalent amount of 1 per cent alcoholic phenolphthalein), and titrate *cold* with standard

TABLE CXI.III

RELATION BETWEEN ACID, LACTONE, ESTER AND SAPONIFICATION VALUES

Saponification Value (Saponifiable Matter)	Acid Value	Free Fatty Acids	
		Free Resin Acids	
	Lactone Value	Free Asphaltous Acids	
		Anhydrides and Lactones	
Unsaponifiable Matter	Ester Value	Neutral Fats	[Glycerol]
		Waxes	Fatty Acids
			Fatty Acid.
		Free Higher Alcohols	Higher Alcohols
			Hydrocarbons

N/10 caustic potash.† As the free acids are neutralized by the alkali, the barium soaps are precipitated, and at the same time the unsaponified substances are thrown out by the water contained in the *N*/10 caustic potash, until at the close of the titration the solution becomes almost clear, rendering the end-point sharp. The acid value is equivalent to the number of milligrams of potassium hydroxide required to neutralize the free acids in 1 g. of the substance.³⁰⁶ A potentiometric method has likewise been proposed for ascertaining the free acids in petroleum products.³⁰⁷

* Bituminous materials with high fusing-points should be fluxed to semi-liquid consistency with a given weight of neutral paraffin oil.

† Prepared by dissolving 5.612 g. pure caustic potash in 500 ml. 95 per cent alcohol, diluting to exactly 1 liter with water at 60° F. and carefully standardizing against sulfuric acid of known strength.

Test 37b. Lactones and Anhydrides ("Lactone Value"). These are determined as follows: (1) Find the acid value (Test 37a), and the ester value (Test 37c) of the original substance; (2) Find the acid value (Test 37a) and the ester value (Test 37c) of a weighed quantity of the free acids liberated from the substance after saponification. If acid and ester values (1) are equal to respective acid and ester values (2), then lactones *only* are present. If acid value (1) is less than acid value (2), and ester value (2) is equal to 0, then glycerides *only* are present. If acid value (1) is less than acid value (2), and ester value (1) is greater than ester value (2), then *both* glycerides and lactones are present.

The true ester value is equal to ester value (1) minus ester value (2); and the true lactone value is equal to ester value (2).

The foregoing results may be checked by finding the acid values of the original substance and the liberated acids. The true ester value equals the acid value of the free acids minus the acid value of the original material. Similarly, the lactone value is equal to the saponification value minus the sum of the acid and ester values.

Test 37c. Neutral Fats ("Ester Value"). The ester value corresponds to the number of milligrams of potassium hydroxide consumed in saponifying esters (neutral fats, otherwise known as triglycerides). If lactones or anhydrides are absent, the ester value may be *calculated* by subtracting the acid value from the saponification value. If lactones and anhydrides are present, then the ester value may be calculated by subtracting the sum of the acid and lactone values from the saponification value.

Test 37d. Saponification Value. The saponification value represents the number of milligrams of potassium hydroxide consumed in the *complete* saponification of 1 g. of the substance. It represents the sum of the acid, lactone and ester values, and is ascertained in the following manner:

Prepare a 5 per cent solution of caustic potash dissolved in equal volumes of 95 per cent ethyl alcohol and 90 per cent thiophene-free benzol or xylol, let stand at least twenty hours, filter through an asbestos filter and standardize against sulfuric acid of known strength.* Saponify 5 g. of the substance † with 50 ml. of this solu-

* Approximately 45 ml. of *N*/ sulfuric acid will be required to neutralize 50 ml. of the 5 per cent caustic potash solution.

† If the substance fuses above 180° F. (R. and B. Method), flux it back to this

tion (added from a calibrated pipette) by boiling over an electric hot plate under a reflux condenser 1 to 4 hours, depending upon the rapidity with which the substance goes into solution. Before disconnecting the flask, wash out the condenser with a few milliliters of neutral alcohol. Evaporate the benzol on a water bath, add 100 ml. water, boil, decant from the residue, add 50 ml. more water, boil, decant and repeat until all the alkali has been removed (tested by adding a drop of phenolphthalein). Combine the extracts, add 20 ml. of 25 per cent barium chloride solution ($\text{BaCl}_2 \cdot 2\text{H}_2\text{O}$), and 3 ml. each of 1 per cent alcoholic phenolphthalein solution and a 3 per cent alcoholic solution of alkali blue 6-B.* Titrate the warm solution with normal sulfuric acid. As the barium hydroxide becomes neutralized, a copious precipitate of barium sulfate forms which renders the end-point distinct. When the color changes, boil, and if necessary run in more sulfuric acid until the color remains green on boiling. Calculate the quantity of caustic potash required for saponification.³⁰⁸

The titration of dark-colored solutions has been proposed, by saturating a filter paper with standard phenolphthalein indicator, then as the end-point is reached during the titration, a streak of the solution is placed on the sensitized paper with a stirring-rod.³⁰⁹

The following results have been reported for the saponification values of petroleum and native asphalts derived from various sources:³¹⁰

Petroleum Asphalts	Saponification Value Mg./g.	Natural Asphalts	Saponification Value Mg./g.
Arkansas.....	0.68	Bermudez (Lake)	0.20
California.....	0.50-3.25	Bermudez (fluxed) ...	11.30
Mexico.....	1.05-1.30		
Russia.....	0.99	Road Oils	
Texas.....	0.46	California.....	0.75 2.75
Venezuela (Quire Quire)	5.70	Midcontinent.....	0.40
Venezuela (Lake).....	1.04	Oklahoma.....	1.04
West Virginia.....	0.56	Pennsylvania.....	0.42
Wyoming.....	0.36		

The foregoing method may be used for dark-colored, difficultly saponifiable substances, such as montan wax. The use of normal fusing-point, or lower, with a neutral mineral oil, which should previously have been run for a blank test.

* Julius Marcusson finds that by using the two indicators together, the end-point of the titration is sharper, being evidenced by a change in color from red to green.

butyl alcohol in place of ethyl alcohol will enable the saponification to proceed more rapidly, on account of its higher boiling point, and is therefore recommended for substances which saponify with difficulty.³¹¹

A potentiometric method has been proposed³¹² for ascertaining the saponification value of asphalts and mixtures of asphalts with vegetable oils, consisting of the following: To 1 g. of the substance add 50 ml. of approximately 7/10 *N.* KOH in ethyl alcohol and 40 ml. c.p. anisole (methylphenylester). Boil under a reflux condenser for one hour, cool and titrate potentiometrically with standard aqueous HCl. A normal calomel electrode and a hydrogen electrode of the usual type are used as reference and indicator electrodes respectively. During the titration two layers will separate, the upper anisole layer containing almost all the asphalt. About 9 minutes are required to attain a steady potential after each addition of acid. The first maximum represents the neutralization of excess alkali, and the last maximum represents the completion of the reaction between the HCl and the alkali in combination. Hence the amount of acid used between the first and last maxima is a direct measure of the combined alkali. By this method, the saponification value of gilsonite was found to be 9.9 and that of Bermudez asphalt 27.5. The foregoing procedure is particularly suitable for mixtures having a dark color, where readings with indicators are difficult to obtain.

Another modification³¹³ consists in dissolving the substance in 1 to 2 parts by weight of white petroleum oil and shaking with anhydrous alcohol KOH for 30 to 60 minutes at 100° C. On cooling to room temperature without agitation, the saponified residue will adhere to the bottom of the flask, whereupon the alcoholic layer containing some of the oil is poured off for titration.

Test 37e. Separation of Saponifiable Constituents. The following procedure has been devised by the author specifically for examining bituminous materials or admixtures of bituminous materials with animal or vegetable oils and fats, since the customary methods do not adapt themselves especially well, due to the formation of troublesome emulsions. The bituminous material is first freed from insoluble constituents, including any mineral matter, by boiling with benzol under a reflux condenser, cooling and filtering

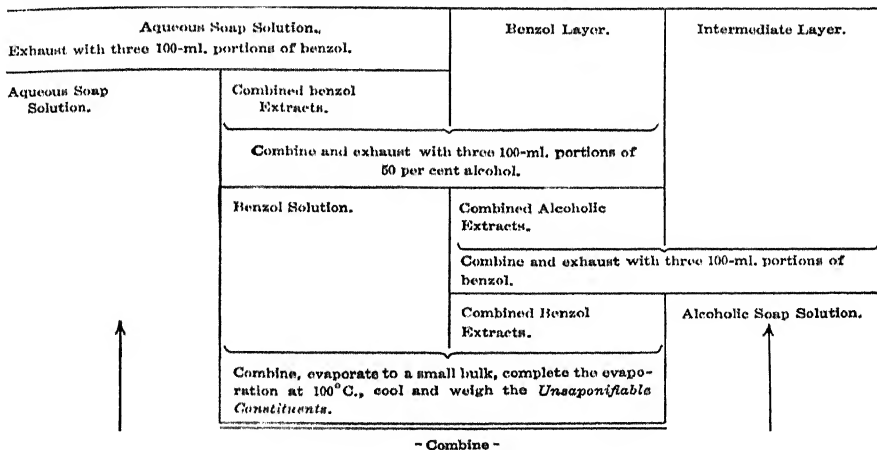
through a Gooch crucible, following the precautions described in Test 21. The insoluble constituents are dried at 100° C. and weighed. Sufficient of the bituminous substance should be taken to yield approximately 5.0 g. of extract. After filtration of the benzol solution, evaporate to dryness on a steam-bath at 105° C., and 5.0 g. of the extract is weighed out and dissolved in benzol, whereupon 50 ml., and 50 ml. of the saponifying liquid added from a pipette. This should consist of a 10 per cent solution of caustic potash, prepared by dissolving 100 g. of anhydrous potash in 500 ml. of 95 per cent ethyl alcohol, and diluting to a liter with 90 per cent benzol. The liquid is allowed to stand overnight to permit any carbonate to settle, and the clear solution decanted. After the saponifying agent is added, the mixture is boiled under a reflux condenser for one-half to one hour, and the contents of the flask, while still warm, poured in a 1000 ml. separatory funnel containing 150 ml. of boiling water and 25 ml. of a 10 per cent solution of potassium chloride. Cool somewhat and add 250 ml. of benzol, agitate vigorously for 5 min., and allow the funnel to rest quietly in a warm place until the solvent separates. If an emulsion forms which refuses to separate on standing, add 200 ml. more benzol and 100 ml. 95 per cent ethyl alcohol and stand in a warm place overnight. This will invariably effect a more or less complete separation of the solvent. From this point on the method is illustrated by the tabular outline on page 1223.

Transpose with dilute hydrochloric acid (10 ml. conc. HCl and 25 ml. water), add a few drops of methyl-orange indicator, making sure that the solution is acid, warm and extract with 100-ml. portions of benzol until the aqueous solution becomes colorless. Separate the aqueous solution containing the glycerol and mineral salts. Evaporate the combined benzol extracts to a small bulk, and then complete the evaporation of the solvent in a weighed crystallizing dish (77 mm. diameter by 40 mm. deep) on a steam-bath until there is no odor of benzol. Finish in an oven at 105° C. for 2 hours. Cool and weigh. The weight equals *the free acids derived from the saponifiable constituents.*

In the case of bituminous materials that are more or less completely saponifiable, the intermediate layer is apt to be absent. In

Saponify as described.

Draw off the aqueous soap solution as completely as possible into another separatory funnel. Decant the benzol layer, leaving the intermediate layer in the original separatory funnel.



this case the process will simplify itself considerably. The foregoing procedure will separate the unsaponifiable constituents in practically an ash-free state.³¹⁴

Test 37f. Examination of Unsaponifiable Constituents. To separate the hydrocarbons, boil 2 g. of the unsaponifiable matter with 4 ml. of acetic anhydride under a reflux condenser for one hour. Add 25 ml. of 95 per cent ethyl alcohol, heat to boiling, decant through an asbestos Gooch crucible, and remove all traces of acetic anhydride by washing with successive portions of warm alcohol. Dry the residue on the Gooch at 100° C. Its weight is equal to the hydrocarbons present.

To separate the higher alcohols (cholesterol), the filtrate from the foregoing is evaporated to dryness, then dissolved in the smallest possible quantity of hot absolute ethyl alcohol and allowed to cool. The cholesterol and phytosteryl (sometimes termed sitosteryl) will crystallize as acetates. Filter and wash with 95 per cent alcohol. Find the melting-point by the capillary-tube method as ordinarily used for pure organic substance. Cholesterol acetate will melt between 114.3 and 114.8° C., whereas phytosteryl acetate will melt above 125° C. Recrystallize several times from hot abso-

lute alcohol and redetermine the melting-point. If the fifth to seventh crop of crystals test below 115-116° C., then phytosteryl is absent.

Cholesterol may also be detected by boiling 1 g. of the substance with 2 ml. of chloroform and 20 drops of acetic anhydride. The solution is allowed to cool and the clear liquid decanted into a porcelain crucible. Then 1 drop of concentrated sulfuric acid is added to the liquid. If cholesterol is present, a violet-pink to reddish coloration will be obtained. (For the behavior of resin acids in the foregoing test, see Test 41.)

Cholesterol indicates the presence of *animal* oils, fats or waxes (such as wool grease), whereas phytosteryl indicates *vegetable* oils, fats or waxes. This test is therefore of value in detecting which class of substances is present in admixture with bituminous material.

Test 37g. Examination of Saponifiable Constituents. If both fatty and resin acids are present, these may be separated quantitatively by the Twitchell-Giladding method, or by the Hans Wolff method, the details of which will be found in standard textbooks.

Test 37h. Glycerol. The presence of animal and vegetable oils or fats (triglycerides) will be indicated by determining the glycerol liberated upon saponification. Certain fatty-acid pitches similarly contain a small percentage of glycerol. This is determined as follows: Saponify 5-10 g. of the substance under examination, weighing exactly, and using 25 ml. of the saponifying agents described in Test 37e. Extract the unsaponifiable constituents with benzol as described, and then transpose the soap solution with a *slight* excess of dilute sulfuric acid (instead of using hydrochloric acid). Warm the liquid and extract the fatty acids with benzol.

Evaporate the aqueous solution to a small bulk, and make slightly alkaline with dilute caustic soda. Cool, dilute to about 100 ml. and determine glycerol by any of the standard methods proposed for this purpose.³¹⁵

ASPHALTIC CONSTITUENTS

The methods which follow have been proposed by Julius Marcussou³¹⁶ for differentiating between native and petroleum asphalts. They also give an insight into the composition of asphalts them-

selves, and in this respect the author regards them of special merit. The value and possibilities of these determinations do not appear to be generally appreciated, but as time goes on they will certainly be recognized. A schematic outline of the procedure is shown in Table CXLIV, in which is included a further refinement proposed by Hans Pöll³¹⁷ for separating the asphaltenes into its two components. Table CXLV gives the results reported from several sources³¹⁸ applicable to typical asphalts.

Table CXLVI shows the effect of the blowing process on a specimen of petroleum asphalt.³¹⁹ It will be observed that the percentage of asphaltenes is increased at the expense of the asphaltic resins and oily constituents. In addition, the particles of carbon coalesce through the abstraction of the protective bodies and become coarser, as is evidenced under the ultramicroscope. The higher asphaltene content results in a higher fusing-point; the decreased content of asphaltic resins decreases the ductility; and the presence of the oily constituents accounts for a low break-point, resulting in the blown product having a great elasticity at low temperatures, together with pronounced rubber-like characteristics.

Test 38a. Free Asphaltous Acids. Dissolve 5.00 g. of the asphalt in 25 ml. benzol by boiling under a reflux condenser. Add 200 ml. of neutral ethyl alcohol, let settle, decant the solution from the pitchy residue, and titrate the former cold with *N*/10 alcoholic sodium hydroxide, using phenolphthalein as indicator. Dilute with an equal volume of water and extract the unsaponifiable constituents by shaking with successive portions of benzol until the extract becomes clear. Evaporate the alcoholic soap solution to a small bulk, liberate the asphaltous acids by acidifying with hydrochloric acid, extract with benzol, evaporate the extract to dryness at 105° C. and weigh. The free asphaltous acids appear as a brownish-black tar-like to resinous mass, soluble in alcohol, benzol and chloroform, but nearly insoluble in 88° petroleum naphtha. When heated to 120–200° C. they are converted into the corresponding anhydrides, and at higher temperatures are transposed into unsaponifiable substances resembling asphaltenes. In ethereal solutions they are precipitated by mercuric bromide. With vanillin-hydrochloric acid they give the same color reaction as aliphatic ketones.³²⁰ Extrac-

TABLE CXLIV. SCHEMATIC OUTLINE OF PROCEDURE TEST 38

Dissolve 5 g. asphalt in 25 ml. c. p. benzol under a reflux condenser with boiling. Add 200 ml. neutral ethyl alcohol, let settle and decant.		Residue:	
<i>Alcoholic Extract:</i> Neutralize with N/10 alcoholic NaOH, using phenolphthalein as indicator; dilute with an equal volume of water; extract the unsaponified constituents with benzol.		↑	
<i>Alcoholic Soap Solution:</i> Evaporate to a small bulk; acidify with HCl; and extract with benzol.	<i>Benzol Extract:</i> Saponify by boiling 1 hour under a reflux condenser with N/1 alcoholic NaOH in the presence of benzol; dilute with an equal volume of water; extract the unsaponified constituents with benzol.	Combine	
<i>Benzol Extract:</i> Evaporate to dryness at 105° and weigh: Free Asphaltous Acids	<i>Alcoholic Soap Solution:</i> Evaporate to small bulk; acidify with HCl; extract with benzol.	<i>Unsaponified Residue (Note A):</i> Dissolve in small quantity (±10 ml.) benzol; pour into 200 ml. 88° petroleum naphtha; let settle for several days; filter through a Gooch crucible; wash with 88° petroleum naphtha.	
<i>Benzol Extract:</i> Evaporate to dryness at 105° and weigh: Asphaltous Acid Anhydrides	<i>Benzol Extract:</i> Evaporate to dryness at 105° and weigh: Asphaltous Acid Anhydrides	<i>Residue:</i> Dry at 105° C. and weigh: Asphaltenes	
		<i>Asphaltenes (Note B):</i> Dissolve in CHCl ₃ ; distribute over 25 g. fullers' earth in a paper thimble; extract with successive portions of cold pyridine.	
		<i>Residue on Fullers' Earth:</i> Extract with a mixture of pyridine and CS ₂ (1:1); distil off CS ₂ and evaporate to a small bulk under vacuum. Add stream of CO ₂ . Add hot water to separate the asphaltic constituents; filter and wash with hot water until free from pyridine.	<i>Pyridine Extract:</i> Evaporate the combined extracts to a small bulk under vacuum in a stream of CO ₂ . Add CHCl ₃ , and wash with HCl to remove all traces of pyridine. Evaporate to dryness under vacuum at 100° C. in a stream of CO ₂ ; and weigh.
<i>Note A</i> —To ascertain only the asphaltenes, asphaltic resins and oily constituents, start the test at this point, using 5 g. of the mineral-free asphalt.		<i>Hard Asphaltic "Carboids" in Asphaltenes</i>	
<i>Note B</i> —The separation of asphaltenes into "Hard Asphaltic Carboids" and "Hard Asphaltic Resins" constitutes an elaboration proposed by Hans Pöll.		<i>Residue on Fullers' Earth:</i> Extract with CS ₂ ; evaporate to dryness at 105° C. and weigh. Asphaltic Resins	
		<i>88° Petroleum Naphtha Solution:</i> Evaporate to 25 ml.; distribute over 25 g. fullers' earth in a paper thimble; extract in Soxhlet with hot 88° petroleum naphtha.	
		<i>Residue on Fullers' Earth:</i> Extract with CS ₂ ; evaporate to dryness at 105° C. and weigh.	<i>88° Petroleum Naphtha Extract:</i> Distil to small bulk; evaporate to dryness at 100° C. under vacuum and weigh.
		Oily Constituents	

TABLE CXLV
ASPHALTIC CONSTITUENTS PRESENT IN TYPICAL ASPHALTS

Type of Asphalt:	Fusing-Point °F.	Penetration 100 g./5 sec.		Ductility at 77° F.	Break-point °F.	Free Asphaltous Acids, %	Asphaltous Acid Anhydrides, %	Asphaltenes, %	Asphaltic Resins, %	Oily Constituents, %
		At 77° F.	At 100° F.							
<i>Natural Asphalts:</i>										
Alberta asphalt sand(a).....	50(c)	Soft	Soft	7	2.0		22.5	24.0	51.5
Val de Travers asphalt(a).....	50(c)	Soft	Soft	7.1	1.3	12.9	33.2	42.2
Trinidad asphalt(a).....	132(r)	4	9	6.4	3.9	37.0	23.0	31.0
Bermudez asphalt (refined).....	135(r)	25	11	3.8	2.0	35.3	14.4	39.6
Fluxed grahamite(b).....	161(r)	0.90	1.28	17.28	30.75	48.50
<i>Residual Oils:</i>										
U. S. Mid-continental petroleum.....	80(c)	Soft	Soft	Soft	0.92	0.46	Trace	25.34	74.59
Californian petroleum.....	81(d)	Soft	Soft	Soft	4.04	34.41	60.71
Mexican (Panuco) petroleum.....	85(d)	Soft	Soft	Soft	13.65	26.64	59.20
Cracking coil (low pressure).....	103(d)	183	Soft	+110	11.56	13.92	74.52
Cracking-coil (high pressure).....	105(d)	150	Soft	+110	14.43	9.32	76.15
<i>Blown Petroleum Asphalts:</i>										
Illinois petroleum.....	133(d)	30	93	27	18.32	28.30	52.80
Venezuelan petroleum.....	140(d)	60	12	-18½	20.0	26.0	54.0
Californian petroleum.....	144(d)	21	70	31	19.16	28.54	52.30
Mexican (Panuco) petroleum.....	144½(d)	40	94	12	16.38	29.93	53.69
Colombian petroleum.....	147½(d)	39	92	14	13.66	36.76	49.58
Venezuelan (Quire Quire) petr'm.....	148(d)	16	47	40	26.97	4.45	68.58
Cracking-coil (low pressure).....	148(d)	40	100	14	26.84	28.26	44.88
Cracking-coil (high pressure).....	148(d)	22½	63	25	22.75	8.24	69.01
Mexican (Panuco) petroleum.....	155(d)	42	91	10	30.25	27.30	41.83
Mexican petroleum.....	192(d)	19	5	1½	30.4	17.6	52.0
<i>Residual Asphalts:</i>										
Kansas petroleum.....	0.0	3.0	24.0	11.0	62.0
Russian petroleum.....	0.0	2.0	15.5	16.1	66.0
German petroleum.....	0.0	4.0	4.4	8.6	83.0
Illinois petroleum.....	102(d)	235	Soft	+110	5.24	35.12	59.49
Venezuean (Quire Quire) petr'm.....	102½(d)	190	Soft	+110	3.19	23.88	70.65
Colombian petroleum.....	107½(d)	189	Soft	+110	6.90	20.40	72.60
Mexican (Panuco) petroleum.....	108(d)	160	Soft	+110	16.40	30.77	52.83
Californian petroleum.....	131(d)	39	+100	26½	4.6	59.0	36.4
Venezuelan petroleum.....	133(d)	34	+100	7	16.0	35.0	49.0
Mexican petroleum.....	134½(d)	38	76	10½	22.0	34.0	44.0
Mexican petroleum.....	160(c)	0.61	Trace	5.81	26.72	65.45
<i>Sludge Asphalt:</i>										
U. S. petroleum (blended).....	180(c)	0.81	1.61	27.01	25.68	44.09

(a) Freed from associated mineral constituents.

(b) Grahamite 15% fluxed with residual oil (from asphaltic petroleum) 85%.

(c) K. and S. method

(d) R. and B. method.

TABLE CXLVI

EFFECT OF THE BLOWING PROCESS ON THE ASPHALTIC CONSTITUENTS

	Fusing-point (K. & S.) °F.	Asphaltous Acids and Anhydrides %	Asphaltenes %	Asphaltic Resins %	Oily Constituents %	Ash %	Total %	Molecular Weight of Asphaltenes	Fusing-point Asphaltenes °F.
Original asphalt	93	1.85	24.2	15.0	55.0	4.3	100.35	2219	313-324
Blown asphalt	219	0.87	47.8	9.4	47.8	4.8	100.27	2560	491-505
" "	272	0.64	48.4	8.4	39.2	4.2	100.84	3140	579-587
" "	304	0.46	53.4	7.8	34.9	4.62	101.18	4225	635-644
" "	336	0.42	57.8	8.0	29.1	4.38	99.79	4690	680-695

tion with *N*/sodium carbonate, followed by acidification of the extract with sulfuric acid, removes the ether-soluble resin acids.³²¹

Test 38b. Asphaltous Acid Anhydrides. In the foregoing test, the unsaponified portion is united with the pitchy substances precipitated by alcohol from the original benzol-alcohol solution. These are saponified by boiling under a reflux condenser for one hour with *N*/alcoholic caustic potash in the presence of benzol, the solution is diluted with an equal volume of water, and the unsaponified constituents extracted with successive portions of benzol. The alcoholic soap solution is then evaporated to a small bulk, the asphaltous acid anhydrides liberated by acidifying with hydrochloric acid, extracted with benzol, evaporated to dryness at 105° C. and weighed. These are very similar in appearance to the free asphaltous acids. On heating to high temperatures, they are converted into unsaponifiable products similar in appearance to the asphaltenes.

Test 38c. Asphaltenes. After separating the saponifiable constituents according to Tests 38a and 38b, the bodies which have not combined with alkali are dissolved in the smallest possible quantity of benzol (not exceeding 10 ml.), and the solution poured into 200 ml. of 88° petroleum naphtha.* The insoluble matter is filtered on a Gooch crucible as described in Test 21, washed with 88° naphtha, dried and weighed. This represents the asphaltenes, which appear as a dark brown to black powder similar to grahamite in characteristics. On heating, asphaltenes do not melt, but swell and decompose into a compact and hard coke. Asphaltenes are supposed

* Of which at least 85 per cent by volume should distil between 35 and 65° C.

to be formed by the addition of oxygen or sulfur to petroleum resins, also by intermolecular changes taking place on heating them in air. They are soluble in benzol, chloroform and carbon disulfide, almost insoluble in alcohol and 88° petroleum naphtha, and sparingly soluble in ether and acetone. The asphaltenes derived from natural asphalts are characterized by the presence of oxygen and a high percentage of sulfur (7 to 13 per cent), and under the influence of light are converted into an insoluble modification. The ratio of carbon to hydrogen is highest in the asphaltenes, diminishing in the remaining constituents.

The asphaltenes and their parent substances, the asphaltic resins, are regarded as saturated polycyclic compounds containing sulfur or oxygen, either of which can replace the other.

Asphaltenes are produced by heating the asphaltic resins, accompanied by a darkening in color and a gradually decreasing solubility in petroleum naphtha. Their specific gravity is greater than unity. The asphaltenes impart hardness to the asphalt, hence the greater the percentage present, the harder will be the asphalt. Asphaltenes and asphaltic resins (Test 38*d*) react with fuming nitric acid forming nitro-addition products, soluble in alcohol and acetone, and forming water-soluble products with alcoholic solutions of alkalies. Concentrated sulfuric acid and fuming sulfuric acid when heated, form addition products which are unaffected by alkalies. They also react with mercuric chloride and ferric chloride in chloroform solutions, forming double salts.

Test 38*d*. Asphaltic Resins. The solution of 88° petroleum naphtha obtained from Test 38*c* is evaporated to about 25 ml., distributed over 25 g. fuller's earth in a paper thimble, and extracted hot in a Soxhlet with 88° petroleum naphtha. Aluminum oxide with a trace of CaO added has been suggested as a substitute for the clay.²²² If the first extract is dark colored, it is concentrated to about 25 ml., poured over more fuller's earth, and the process repeated. The extract should have a straw or light yellow color. The asphaltic resins are adsorbed by the fuller's earth, from which they may be extracted by carbon disulfide, evaporated to dryness at 105° C., and weighed. These form the first stage in the conversion of petroleum hydrocarbons into asphaltenes, which consist of solid, reddish-brown to brownish-black substances fusing below

100° C., completely soluble in 88° naphtha, chloroform, carbon disulfide, benzol, but only sparingly soluble in hot or cold acetone. When adsorbed by fuller's earth they become insoluble in 88° petroleum naphtha and are no longer soluble therein after removal from the fuller's earth (probably due to oxidation by air while in contact with the earth). The asphaltic resins are formed by heating the oily constituents for some time to 120° C. accompanied by a darkening in color and absorption of atmospheric oxygen. High content of asphaltic resins produces great ductility and high break-point of the asphalt.

Sulfur dissolves in the resins when heated, with the liberation of hydrogen sulfide. The resins derived from coal-tar pitches when heated with strong sulfuric acid form water-soluble sulfo-compounds, whereas petroleum resins form insoluble products. Their specific gravity is in the neighborhood of unity.

Test 38e. Oily Constituents. The 88° petroleum naphtha extract from the fuller's earth in Test 38*d* is distilled to a small bulk, and evaporated to dryness at 100° C. under vacuum until the odor of petroleum naphtha is no longer apparent. The oily constituents remaining as residue are weighed. These appear as a viscous oil, and constitute the most inert bodies contained in asphalts. They are optically active and usually fluorescent, show a specific gravity of less than unity, and have about the same iodine value as viscous lubricating oils derived from petroleum. In general they may be said to be composed of saturated and unsaturated hydrocarbons containing cyclic compounds, and containing small percentages of sulfur, nitrogen and oxygen.²²³

As a general rule, the softer the asphalt, the larger will be the percentage of oily constituents. Marcusson reports that Trinidad petroleum contains 42.5 per cent of oily constituents, Trinidad asphalt 17 to 19 per cent (figured on the crude dry substance containing the mineral ingredients) and grahamite 2 to 3 per cent.

The following modification has been described for the separation of "asphaltic resins" and "oily constituents" in the presence of wax:²²⁴

Weigh 10–15 g. asphalt into a 2-liter flask. Add 50 volumes n-pentane per volume asphalt, in two additions. The first addition is heated or refluxed to disperse the asphalt, whereupon the remain-

der of the pentane is introduced. Cool to 77° F. for 1 hour and filter through a Büchner funnel. Distil the filtrate to 150 ml., evaporate dry over a water bath and remove the last traces of pentane under vacuum. Weigh and calculate the percentage soluble in pentane.

Boil the residue with 6 times its weight of isobutyl alcohol under a reflux condenser for 15 minutes, cool to 150° F., add NH_4OH solution (27 per cent) to an amount of 3 per cent by weight of the isobutyl alcohol present. Cool to 130° F., let stand 10 minutes and centrifuge 3–5 minutes at 130° F. Decant the solution from the centrifuge tube through a filter into a weighed 250-ml. Erlenmeyer flask. Wash out the centrifuge with 3 per cent of NH_4OH in isobutyl alcohol at 130° F. The solution containing the "oily constituents" and "associated waxes" is distilled under vacuum and the residue weighed.

To the residue of oils and waxes add 10 parts by weight of a solvent containing 3 vols. of acetone and 2 vols. of methylene chloride. When dissolved, cool to 32° F. and filter off the precipitated wax in a funnel maintained at 32° F. Wash with cold solvent. Dry and weigh the recovered wax.

Calculate the oily constituents by difference, or else recover by evaporating the filtrate under vacuum and weigh. In this manner, the resins, oils and waxes are recovered quantitatively and may be examined further for other characteristics.

A modification of this test has been proposed for completely separating waxes from the resin fractions of paraffinic-base asphalts,^{324a} involving the precipitation and removal of: (1) the insolubles with hexane at 25° C. (77° F.), (2) the hard resins with a solution of cyclohexane-isobutyl alcohol at 37.8° C. (100° F.), (3) the waxes with an acetone-methylene chloride solution at 0° C. (32° F.), and (4) the soft resins with isobutyl alcohol at 37.8° C. (100° F.). The portion soluble in isobutyl alcohol is classed as "oily constituents."

The oily constituents derived from natural asphalts are characterized by the presence of small percentages of solid paraffins (usually less than 1 per cent) and by the fact that they appear fluid and sticky at 20° C., whereas those derived from petroleum asphalts appear non-fluid and of a consistency of vaseline. They impart

softness to the asphalt, thus counteracting the hardening properties of the asphaltenes.

Test 38f. Short Method for Determination and Recovery of Difference Resins, Resins and Oily Constituents. The following procedures have been formulated, depending upon whether the "resin mixed with difference resins," or the "resins" alone are required for further investigations (e.g., accelerated weathering tests). The asphaltic components involved in these tests are differentiated as follows:

Asphaltenes: that portion of the asphalt which is insoluble in ethyl ether (likewise insoluble in n-pentane).

Difference Resins: that portion of the asphaltenes which is soluble in ethyl ether, but is insoluble in n-pentane.

Resins: that portion of the n-pentane-soluble asphalt constituents, which when mixed with fuller's earth remains insoluble in n-pentane, but in turn dissolves in ethyl ether.

Oily Constituents: that portion of the asphalt which is soluble in n-pentane when the asphalt is mixed with fuller's earth.

Both methods have the virtue of simplicity, speed of performance, and the small quantity of solvents consumed.

Method I. Melt 10 g. asphalt and mix with 35 g. fuller's earth passing a 100-mesh sieve, and 20 g. fuller's earth passing a 30-60-mesh sieve. Transfer to a paper thimble (4.3 mm. diam. by 123 mm. high) and plug the top with cotton. Extract in a Soxhlet with 300 ml. n-pentane (C_5H_{12}) for 8 hours at a rate of one siphon overflow every 6 minutes. Distil the extract to a low bulk and complete the evaporation of the pentane, first over a steam-bath and finally in an oven at $105^{\circ}C$. The weight of the residue equals the "oily constituents." Then extract the residue in the thimble with ethyl ether in the same manner as described for oily constituents. Evaporate the ether extract to dryness as before, to recover the mixture of "difference resins with resins." Method I is summarized in Table CXI.VII.

Method II. Weigh 10 g. asphalt and transfer to a tared Soxhlet thimble. Extract with 300 ml. n-pentane (C_5H_{12}) as in Method I. Dry the residue in the thimble on a steam bath and then in an oven at $105^{\circ}C$. Weigh the "asphaltenes" plus the "dif-

ference resins" (the latter may be separated by extracting with ethyl ether).

Distil the pentane extract to about 25 ml. in a 600-ml. beaker, add 35 g. of fuller's earth which passes a 100-mesh sieve, and thoroughly mix with a steel spatula. Then add 20 g. of fuller's earth which passes a 30-60-mesh sieve, and mix the mass to make it more porous. Transfer the mixture (before it becomes too dry

TABLE CXLVII

METHOD OF RECOVERING RESINS MIXED WITH DIFFERENCE RESINS

Method I—Recovers "Resins with Difference Resins"

Asphalt mixed with fuller's earth
Extracted with pentane

Insoluble Residue: Asphaltenes + Difference Resins + Resins + fuller's earth Extract with ethyl ether			Soluble Portion <i>Oily Constituents</i>
Insoluble Residue: Asphaltenes + fuller's earth Extract with CS ₂		Soluble Portion: Difference Resins + Resins Extract with pentane	
Insoluble Residue: fuller's earth	Soluble Portion: <i>Asphaltenes</i>	Insoluble Residue: <i>Difference Resins</i>	

and dusty) to a paper thimble (43 mm. diam. by 123 mm. high), plug with cotton, and extract in a Soxhlet with n-pentane for 8 hours as in Method I. Distil off the major portion of the pentane from the extract in a tared beaker, allow the balance to evaporate at room temperature, and finally heat over a steam bath until the odor of pentane has disappeared, followed in an oven at 105° C. for 1 hour. Cool in a desiccator and weigh the "oily constituents." Extract the residue in the paper thimble with ethyl ether in the same manner as described for the oily constituents, until the washings are colorless. Evaporate the ether extract to dryness in a tared beaker, heat in an oven at 105° C. for 1 hour, cool in a desiccator, and weigh the "resins."²²⁵ Method II is summarized in Table CXLVIII.

TABLE CXLVIII
METHOD OF RECOVERING RESINS ALONE

Method II -Recovers "Resins"

Asphalt (in original state)
Extract with pentane

Insoluble Residue: Asphaltenes + Difference Resins Extract with ethyl ether		Soluble Portion: Resins + Oily Constituents Mix with fuller's earth Extract with pentane	
Insoluble Residue: <i>Asphaltenes</i>	Soluble Portion: <i>Difference Resins</i>	Insoluble Residue: Resins + fuller's earth Extract with ethyl ether	Soluble Portion: <i>Oily Constituents</i>
		Insoluble Residue: fuller's earth	Soluble Portion: <i>Resins</i>

DIAZO REACTION

This test is used for identifying bituminous substances carrying phenols, including wood tar and wood-tar pitch, shale tar, peat and lignite tars and pitches, bone tar, bone-tar pitch and the various coal-tar pitches. Oil-gas and water-gas tars and pitches contain but a trace of phenols and hence give but a slight diazo reaction.

Test 39. Graefe's Method. This reaction as devised by Edmund Graefe.²²⁶ It is carried out by boiling 2 g. of the bituminous substance with 20 ml. *N/* aqueous caustic soda, for approximately five minutes. After cooling, the liquid is filtered. If the filtrate is dark colored, it may be lightened by shaking with finely pulverized sodium chloride and filtering. It is then cooled in ice to 10° C., and a few drops of freshly prepared diazobenzolchloride solution added (prepared by dissolving 1 g. aniline chlorhydrate in 10 ml. water and 3 ml. 25 per cent hydrochloric acid, and then adding, drop by drop, a saturated solution of 0.5 g. sodium nitrite in water). To avoid the preparation of diazobenzolchloride (which does not keep), the use of paradiazobenzil-sulfonic acid ($C_6H_4 \cdot N_2SO_3$) may be substituted, since it is a more stable chemical and will keep

well in stock. If phenols are present, a more or less fugitive red coloration will result, sometimes accompanied by a reddish precipitate.

Assuming that the bituminous substance gives the diazo reaction, the question will often arise whether the product is a straight-distilled pitch, or an asphalt "cut-back" with a high boiling-point distillate containing phenolic bodies, derived from coal tar, lignite tar, etc. Julius Marcusson has worked out a method applicable under these circumstances,³²⁷ which consists in dissolving 10 g. of the bituminous substance in 15 ml. of benzol, and pouring the solution into 200 ml. of 88° petroleum naphtha. The resulting precipitate is filtered and washed with petroleum naphtha and dried. It is then boiled for fifteen minutes with $N/2$ alcoholic caustic potash under a reflux condenser to extract the phenols. The liquid is cooled and filtered, the alcohol evaporated, and the residue dissolved in water. Sodium chloride is added to clarify the liquid and remove any substances imparting a dark color, the solution is filtered and the filtrate treated for the diazo test described above. If a straight-distilled pitch containing phenols is present, a positive reaction will be obtained. If the original substance gives the diazo test, but the residue treated in the above way does not, then the admixture of high boiling-point oils containing phenolic bodies with a substance free from phenols (e.g., asphalts, etc.) is established. It is claimed that the presence of as little as 10 per cent of pitch containing phenols may be detected in this manner.

Where bituminous substances contain calcium carbonate, the phenolic bodies present combine with the lime, forming insoluble calcium phenolate which yields but a faint diazo reaction. However, on treating such substances with a solvent in the presence of hydrochloric acid, the calcium phenolate is decomposed, and the diazo reaction becomes much more delicate.

A still more sensitive test for ascertaining the presence of phenols (e.g., tars or pitches) in asphalts consists in the following: A reagent is prepared by dissolving 0.2 g. paranitraniline in 20 ml. water and 5 ml. 20 per cent sulfuric acid, whereupon 0.3 g. sodium nitrite are added. Hard asphalts (3 to 4 g.) are ground in a mortar with 25 ml. water made alkaline with NaOH; then filtered and the filtrate acidified with H_2SO_4 and 3 drops of the foregoing re-

agent added. The solution is finally made alkaline with NaOH, whereupon the presence of phenols will develop a more or less intense red coloration. It is claimed that the presence of 1 per cent of tar or pitch may thus be detected, inasmuch as a 1:25,000 solution of phenol will produce a blood-red color, and a 1:250,000 solution a pinkish-red color. Pure asphalts, on the other hand, yield a light yellow color or leave the reagent unchanged.

The Dutch Standardization Committee³²⁸ have adopted a combination of paradiazobenzol-sulfonic acid with Millon's reagent for detecting the presence of phenols in substances containing colophonium, as follows: 10 g. of the substance are boiled 20 minutes with 25 ml. *N.* aqueous NaOH and filtered. To 5 ml. of the filtrate add a few drops of paradiazobenzol-sulfonic acid. If no red coloration results, phenol (tar or pitch) is absent, but if a coloration occurs, tar or pitch may be present, but it may also be caused by colophonium. In this event, the remainder of the filtrate is almost neutralized with nitric acid and evaporated to 5 ml. Then add 5 ml. freshly prepared Millon's reagent and place in a glass beaker with boiling water. If no coloration results within half an hour, the mixture is free from phenols.

ANTHRAQUINONE REACTION

The anthraquinone reaction is used for detecting anthracene in tar products produced at high temperatures, including oil-gas tar and pitch, water-gas tar and pitch, and the various coal-tar pitches. This test is therefore valuable for purposes of identification.

Test 40. Conventional Method. The tar or pitch is first subjected to distillation in accordance with the retort method (Test 16*b*), the offtake and condensing tube being kept warm to prevent the accumulation of any solid distillate. The distillate passing over between 270 and 355° C. is caught separately and examined for anthracene in the following manner. The fraction is heated until it is thoroughly fluid to secure a uniform sample, and 5 g. weighed out, while hot. After cooling, 10 ml. of absolute ethyl alcohol are added, the solids allowed to crystallize and the liquid decanted. One to 2 g. of solid substances containing the anthracene are dried on a water-bath, transferred to a 500-ml. flask connected with a

return condenser, 45 ml. of glacial acetic acid added, and the contents boiled for two hours. The following mixture is then added drop by drop through a separatory funnel, viz.: 15 g. of anhydrous chromic acid dissolved in 10 ml. of glacial acetic acid, and 10 ml. of water. The boiling is continued for another two hours, the flask cooled, and 400 ml. cold water added. This treatment oxidizes the anthracene to anthraquinone, which on cooling separates as a solid mass. This is filtered, washed with hot water, then with a hot 1 per cent solution of caustic soda and again with hot water. The residue of anthraquinone is then dried and its weight multiplied by 0.856 to obtain the corresponding weight of anthracene. From 0.25 to 0.75 per cent of anthracene is found in coal tars, and a correspondingly larger percentage in coal-tar pitches.

A color reaction for establishing the presence of anthracene consists in boiling for $\frac{1}{2}$ to 1 hour the crystals of anthraquinone (1 part) with zinc dust (2 parts) and 50 per cent NaOH solution (30 parts), whereupon an intense red colored solution is obtained, which on filtering in contact with air becomes decolorized.

LIEBERMAN-STORCH REACTION

Test 41. Colorimetric Method. This is a rapid qualitative test for detecting the presence of rosin,³²⁰ rosin oil, or cholesterol. One gram of the substance is dissolved in 3 ml. of acetic anhydride at a gentle heat, cooled and the clear liquid decanted into a porcelain crucible. Add 1 or 2 drops of sulfuric acid sp. gr. 1.53 (containing 62.53 per cent of pure sulfuric acid, prepared by diluting 34.7 ml. of concentrated sulfuric acid with 35.7 ml. of water). Rosin and rosin oil will produce a fugitive violet coloration turning to a brown, whereas cholesterol will produce a fugitive rose color turning rapidly to a dark green. If rosin or rosin oil is present in conjunction with cholesterol, the test becomes valueless.

The following two methods have been standardized for the detection of resin, etc.:³²⁰

*Method I. Test for Rosin (Lieberman-Storch Method).—*Take 0.1 to 0.2 g. of the material and heat gently with about 15 ml. of acetic anhydride. Cool, filter or decant the clear solution. Place a few drops of the clear solution on a white porcelain cru-

cible cover, and add 1 drop of sulfuric acid (34.7 ml. sulfuric acid, specific gravity 1.84, and 35.7 ml. of water), so that the acid will mix slowly with the filtrate. If rosin is present, a characteristic fugitive violet color develops immediately.

Method II. Test for Rosin (Halphen-Hicks Method for Shellac, Damar Resin, etc.)—

Reagent:

Solution (a): 1 part by volume of phenol dissolved in 2 parts by volume of carbon tetrachloride.

Solution (b): 1 part by volume of bromine dissolved in 4 parts by volume of carbon tetrachloride.

Dissolve a small quantity of the material to be tested in from 1 to 2 ml. of solution (a). Pour the solution into one of the cavities of an ordinary porcelain color-reaction plate until it just fills the depression; a portion of the solution will soon be seen to spread out on the flat part of the plate a short distance beyond the rim of the cavity, unless too much of the carbon tetrachloride has been lost through evaporation during the process of solution, when a drop or two more should be added to produce the spreading effect above referred to. Then immediately in an adjacent cavity place 1 ml. or more of solution (b) so that the bromine vapors evolved will impinge upon the surface of the solution in the other cavity. Sometimes it is necessary to blow a gentle current of air in the proper direction to accomplish this satisfactorily or both cavities may be covered by a watch crystal of suitable size. The development of a violet coloration, best observed upon the flat portion of the test plate, indicates the presence of rosin.

Fossil resins (copals, etc.) also fatty-acid pitches give a permanent brown color and do not interfere with the foregoing test. Linseed, cotton-seed, china-wood and corn oils give a permanent greenish-brown coloration, whereas palm oil, bone tar, and crude olein give a permanent brownish-yellow coloration.

Highly heated rosin and rosin esters do not produce the characteristic color. The presence of rosin may be established with certainty by separating the rosin acids as follows: dissolve 5 g. of the substance in 150 ml. ether, filter, and shake the filtrate twice with 250-ml. portions of 1 per cent aqueous ammonium carbonate solu-

tion and then with 3 portions of 200 ml. each of 1 per cent caustic soda solution. Acidify the extract with hydrochloric acid, and extract the liberated resin acids with ether. Evaporate to dryness and dissolve the residue in 10 parts by weight of petroleum naphtha. Rosin is present if the violet color is produced when tested as above.

CHAPTER XXXIII

EXAMINATION OF BITUMINOUS SUBSTANCES

COMBINED WITH DISCRETE AGGREGATES

Products falling into this class include native and artificial mixtures of bituminous matter with discrete aggregates, viz.: bituminous macadam pavements, bituminous concrete pavements, sheet asphalt pavements, asphalt block pavements, asphalt mastic floorings, bituminous expansion joints (containing mineral matter but not felt), pipe-sealing compounds, molding compositions and products used for electrical insulation.

(A) PHYSICAL TESTS OF FINISHED PRODUCT

(1) PAVING COMPOSITIONS, ASPHALT MASTIC, BITUMINOUS GROUTS, PIPE-SEALING COMPOUNDS, ETC.¹

Test 42. Specific Gravity. This is determined as described in Test 7, except that a larger specimen shall be taken, preferably cut as a cross-section of the completed material, and without the application of heat. The results express the "apparent specific gravity" of the composition, including any voids that may be present.

Test 43. Voids.² This test is adapted for testing compressed paving mixtures, and supplements Test 7f, which may also be used for the purpose. The voids may be ascertained if the specific gravity and the per cent by weight of each constituent are known, as well as the apparent specific gravity of the mixture. Thus, if w_1 , w_2 and w_3 represent the percentages by weight of sand, filler and asphalt, respectively, in a given mixture; and g_1 , g_2 and g_3 their specific gravities, then the theoretically maximum density (D) of the composition (if entirely free from voids) may be calculated from the following formula:

$$D = \frac{100g_1g_2g_3}{w_1 + w_2 + w_3}$$

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The apparent specific gravity (d) of a test specimen having been ascertained, as in Test 42, the per cent of voids (v) may be calculated as follows:

$$v = \frac{100(D - d)}{D}$$

If w_0 = per cent by weight of total aggregate mixture

and g_0 = specific gravity of total aggregate mixture

Then $v = 100 - d \left(\frac{w_0}{g_0} - \frac{w_s}{g_s} \right)$

The per cent of voids (v_m) in the mineral aggregate of a compressed paving mixture (excluding the asphalt itself) is calculated from the expression:

$$g_m = d(1 - w_s)$$

Where g_m equals the apparent specific gravity of the mineral aggregate. The maximum theoretical density of the mineral aggregate (D_1) may be calculated from:

$$D_1 = \frac{\frac{w_1}{g_1} + \frac{w_2}{g_2}}{\frac{w_1}{g_1} + \frac{w_2}{g_2}}$$

and

$$v_m = \frac{D_1 - g_m}{D_1}$$

Test 44. Resistance to Moisture. Various methods have been suggested from time to time for ascertaining the water absorption of paving materials.³ It is recognized that all pavements absorb more or less moisture, but no standard method has been proposed for this purpose. Clifford Richardson suggests the use of cylinders of the same dimensions as used for the impact test, namely 1.25 in. in diameter, by 1 in. high, having the greatest possible density, which in the case of surface mixtures for sheet asphalt pavements, will weigh about 50 g. They are immersed in water for three months, and the gain in weight noted at various intermediate periods.

Test 44a. Swelling Effects of Water. The following procedure has been standardized:^{3a}

These methods provide for the determination of the swell characteristics of dense-graded bituminous mixtures and aggregates used in such mixtures. Method *I* provides for the determination of the swell characteristics of aggregates using the actual grading and bituminous binder intended for a particular project, or for the determination of the swell characteristics of bituminous mixtures as produced during construction. Method *II* provides for the classification of all dense-graded aggregates on a uniform basis, regardless of the type of bituminous binder intended for use. It furnishes a uniform basis of comparison for the characteristics of the aggregates only.

The apparatus required consists of the following:

(a) Mixing bowls or pans and a mixing spoon or paddle of convenient sizes and shapes.

(b) Molds: The molds used in Method *I* shall be bottomless steel cylinders approximately 4 in. inside diameter and 5 in. high, machine finished on each end. The molds used in Method *II* shall be bottomless steel cylinders approximately 3 in. inside diameter and 2 in. high, machine finished on each end.

(c) Base Plates: Flat, machine finished base plates approximately 5 in. square shall be provided for Method *I* and 4 in. square for Method *II*.

(d) Tamper: The tamper for preliminary compaction of the specimen shall be a suitable steel tamper with a striking face of 2 in. diameter, weighing 5 ½ lbs., and equipped with suitable guides for controlling the direction and height of the drop.

(e) Compression Plungers: Compression plungers shall be steel cylinders, 4 in. high for Method *I*, and 2 in. high for Method *II*. The cylinders shall be of such a diameter as to provide a sliding fit within the corresponding molds.

(f) Compression Device: The machine used shall be any type capable of exerting and indicating the required load. For tests in the field a rigid frame and a hydraulic jack equipped with a suitable gage may be satisfactory.

(g) Measuring Device: A dial micrometer reading to 0.001 in. shall be attached to a support in such a manner that changes in the elevation of the top of the specimen at its center may be read. The support shall rest on the top edge of the mold and be so designed that removal and replacement of the measuring device shall cause no change in the indicated reading.

Method I: The bituminous binder shall be the same as that which is to be used in construction of the project.

Method II: The bituminous binder shall be type SC-2 liquid asphalt and shall be from a standardized lot which is used for all tests made in accordance with Method II. The asphalt shall have a naphtha xylene equivalent of less than 15 per cent as determined by the Standard Method of Spot-test of Asphaltic Materials, and shall be from a source which will allow the replenishment of the supply with oil of similar characteristics. (Note)

NOTE.—As oils of the same xylene equivalent but from different sources will affect the swell characteristics by somewhat different amounts, an ample supply of standardized SC-2 oil should be maintained to secure uniform test conditions.

Procedure, Method I. A representative sample of the aggregate as prepared for use, weighing 1,000 g., shall be placed in the mixing bowl and heated in an oven to the temperature necessary for obtaining a uniform mix with the bituminous binder. It shall be combined while hot with the necessary amount of bituminous binder to give a satisfactory mixture. The resulting mix shall be cured in an oven at a temperature of 140° F. (60° C.) for a period of 20 hrs. After the curing period, the mixture shall be compacted in the mold by means of 50 blows of the tamper. The blows shall be distributed uniformly over the specimen. After manual compaction, the mold shall be reversed so that the tamped surface becomes the bottom of the specimen. The plunger shall then be placed on top of the specimen and a total load of 25,000 lbs. applied slowly and maintained for 1 min. before releasing. The plunger shall be removed and the specimen allowed to stand for 1 hr. before measuring. When other than laboratory prepared bituminous mixtures are to be tested, 1,000 g. of the mix as received shall be heated and compacted as above.

Procedure, Method II. A quantity of material passing the No. 3 sieve, sufficient to yield approximately 400 g. of mixed material which will pass a No. 10 sieve, shall be placed in the mixing bowl and heated in an oven at 221 to 230° F. (105 to 110° C.) to uniform temperature. It shall then be mixed with the amount of the standard SC-2 liquid asphalt necessary to give a satisfactory mixture. The resulting mixture shall be cured at room temperature for a period of 20 hrs. The sample shall be screened on a No. 10 sieve and all particles retained on the sieve discarded. Approximately 400 g. of the material passing the No. 10 sieve shall then

be compacted in the mold to a thickness of approximately $1\frac{3}{4}$ in. by means of 30 blows of the tamper. The blows shall be distributed over the entire surface of the specimen. The plunger shall then be placed on top of the specimen and a total load of 15,000 lbs. applied slowly and maintained for 1 min. before releasing. The plunger and any loose material in the mold shall be removed, and melted paraffin poured over the surface of the specimen to effect a watertight seal. The mold and specimen shall be removed from the base plate and inverted so that the surface which was against the base plate will be the top of the specimen. The specimen is now ready for measuring.

Measuring, Methods I and II. A perforated metal disc fitting loosely in the mold shall be placed on the specimen. The measuring device shall be placed on the top edge of the mold and the elevation of the top center of the specimen recorded, using the elevation of the edge of the mold as the datum plane.

Immersion, Method I. The mold shall be filled with water to a depth of 2 in. above the top of the specimen and maintained at that level for a period of 24 hrs. At the end of this period, the elevation of the top of the specimen shall again be measured.

Immersion, Method II. The mold shall be immersed for a period of 24 hrs. in a pan of water of such depth that the top of the specimen shall be covered by 2 in. of water. At the end of this period, the mold shall be removed from the water and the elevation of the top of the specimen measured as before.

The difference between the original elevation and the final elevation shall be considered to be the swell and shall be recorded to 0.001 in.

The following shall be reported:

- (a) Method used (*I* or *II*).
- (b) Type of bituminous binder used.
- (c) Percentage of bituminous binder used.
- (d) Swell.

The following may also be reported:

- (e) Actual thickness of compacted specimen before immersion.
- (f) Swell, expressed as a percentage of the actual thickness of the compacted specimen before immersion.

Test 45. Effect of Water on Adhesion. The following method ⁴ has been proposed to determine the adhesion of asphalts to various types of mineral aggregate when subjected to the influence of water. The paving mixture is warmed until soft and then broken up with a spoon or spatula while cooling. When cool, 50 g. are placed in a 250-ml. pyrex flask to which 175 ml. of water at 140° F. are added. The flask is then corked and rotated for 1 hour at the rate of 39 to 40 revolutions per minute while immersed in a water bath maintained at 140° F. The flask is then removed and the sample examined to ascertain the extent to which the asphalt has been washed off the mineral aggregate, which ordinarily may be easily observed. In cases where this is not clearly visible, uncovered grains may be collected at the bottom of the flask, by rotating it a few times by hand, whereupon all the uncovered grains will congregate together.

Modifications of the test involve:

(a) Soaking the mixture in distilled water at 140° F. for 18 to 20 hours.

(b) Boiling the mixture in distilled water for 1 minute (after having raised the temperature to the boiling-point in 6 minutes).

(c) Allowing a briquette of the coated aggregate to soak in distilled water at room temperature until disintegration takes place.

(d) Boiling the mixture in sodium carbonate solutions of varying strengths, and recording the strength of the solution which causes a separation between the asphalt film and mineral aggregate, as a quantitative measure of the adhesion.

The adhesion between asphalt and mineral fillers is considered good when water, etc., do not cause a separation, and poor when the asphalt draws away from the filler. In the former case the filler is said to be hydrophobic, since it is wetted by the asphalt more readily than by the water, and in the latter case, the filler is said to be hydrophilic. Good fillers, therefore, are hydrophobic in character.

Another procedure to measure the adhesion of bituminous substances to mineral aggregates consists in subjecting the paving mixture to attrition under water in a road-testing machine by which weighted rubber-tired wheels are caused to rotate in a trough (similar to a chilean mill).⁵

The following fillers were found to have a greater affinity for water than for asphalt, viz.: silica (impure), infusorial earth, clay, iron oxide, aluminum oxide (bauxite), impure limestone, calcined magnesia, gypsum, barytes, lithopone, granite containing feldspar, and certain kinds of slag. The following fillers have a greater affinity for asphalt than for water, viz.: pure silica (e.g., crushed Ottawa sand), pure limestone, hydrated lime, powdered limestone rock-asphalt, and calcined coal-mine waste. Even with pure limestone, Bermudez asphalt adheres much better than do petroleum asphalts. Natural limestone rock-asphalts from Texas, Sicily, Germany, France and Alabama adhere very much better than do petroleum asphalts.

Examination corresponding to Tests 46, 47, 49, 50, etc., may also be conducted at low temperatures, as for example: at 32° F. by cooling with ice; at 0° F. by using a mixture of ice with salt or calcium-chloride; or at temperatures as low as -90° F. by using dry ice.^a

Test 46. Hardness. The following test has been proposed⁷ for this purpose: test pieces are prepared in the form of cylinders 2 in. in diameter and 2 in. high, either by cutting from the solid mixture by means of a carborundum saw, or by molding the material, warmed to 130-140° F. under sufficient pressure (about 12,000 lb. per sq. in.), preferably in a mold with a double plunger acting on top and bottom of the specimen, so that after compression it will have the same gravity as the original mixture. After molding, the test pieces are allowed to age at room temperature for 7 days. The hardness is defined as the depth expressed in hundredths of a cm., to which a steel rod of 6.35 mm. diameter, with the end cut off square, will penetrate the test piece in 60 seconds at 77° F., under a load of 100 kg. per sq. cm. The testing machine is illustrated in Fig. 339. A vertical rod has attached to its lower end a smaller steel rod 6.35 mm. in diameter. The horizontal beam is pivoted near its center, and counter-balanced by the weight shown at the left of the illustration. The test specimen is maintained in a water-bath at 77° F. The load is applied by placing the right-hand weight at a predetermined position on the beam. The distance penetrated is indicated by a vernier gear attachment, which records

the depth in hundredths of a cm. The right-hand weight may be fixed at different positions to give different loads, and the temperature may be varied, as desired. An average of 6 separate readings is taken, being careful to keep away from the edge of the specimen. A similar procedure consists in measuring the distance in which a

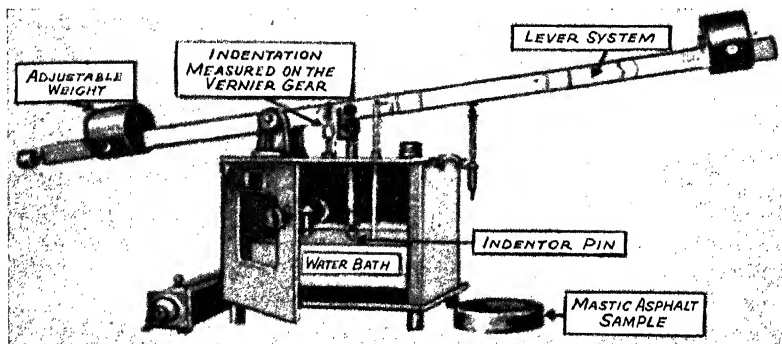


FIG. 339.—Hardness or Resistance to Indentation Carried Out Directly on the Finished Mastic Asphalt.

Consists essentially of an electrically heated oven the interval temperature of which can be controlled accurately. The vertical movement of the indenter pin may be measured accurately by a vernier gear. A known load can be applied to this indenter pin by means of a lever system. The load is applied for a period of one minute.

steel ball will penetrate into the substance under a given loading and temperature.⁸

Sundry other hardness (i.e., indentation) tests have been proposed from time to time.⁹

Test 46a. Flexural Strength. Beams are molded in a steel mold, 8 in. long, 2 in. wide and 1½ in. deep by heating the ingredients to 350° F., suitably mixing them together in the required proportions, and filling the mold at 250° F. as described in Test 47. The beam is brought to the desired test temperature and supported on two rollers set at a 6 in. span and the load applied at the center as described in Test 54. The modulus of rupture is calculated, and the deflection is measured by a suitable attachment.

Test 46b. Workability Test. Used for testing bituminous enamel as follows:¹⁰

The enamel and primer shall be applied to a clean smooth, soft steel plate, 30 cm. by 30 cm. by 1.6 mm. (12 by 12 by $\frac{1}{16}$ in.) in the manner best adapted for its use, leaving a narrow uncoated border about the edge of the plate. The plate shall first be coated with the primer and when this has dried to a tacky state, the enamel shall be spread at not exceeding 350° F. in a coating 3 to 5 mm. ($\frac{1}{8}$ to $\frac{3}{16}$ in.) thick. The spreading and working qualities, together with the character of the resulting surface, shall determine its workability.

Test 47. Resistance to Displacement. The following modifications have been proposed:

(1) *For Testing Paving Mixtures.* The following test has been proposed for examining fine-aggregate paving mixtures (e.g., the wearing-course of sheet-asphalt pavements),¹¹ and has also been adapted for testing coarse-aggregate paving mixtures.¹² This test has also been termed an "extrusion" or "stability" test.

The procedure when testing fine-aggregate mixtures consists in weighing the dry sand and filler to 0.1 g., heating to 350° F. and mixing thoroughly. The melted asphaltic binder heated to 350° F. is then weighed to 0.1 g. and thoroughly mixed with the aggregate by means of a spatula. Approximately 100 g. of the completed mixture are weighed into a cylindrical forming mold 2 in. in diameter and 4 in. high. The mold and contents are heated to 250–260° F. as determined by the thermometer used for stirring the mixture. Remove and insert a cylindrical plunger into the mold, and then tamp by first giving 60 blows with a tamper weighing 500 g., followed by 15–20 blows with a tamper weighing 1,400 g. The mold and contents are placed in a compression machine to which is applied a load of 3,000 lb. per sq. in. and the briquette allowed to cool under pressure. The compressed briquette should measure 2 in. in diameter and approximately 1 in. in height. Let stand at room temperature over night and then maintain in a water-bath at 140° F. for one hour before testing. Two such briquettes should be prepared, each specimen being tested by placing it top end down in the testing mold and inserting the plunger as illustrated in Fig. 340. A circular washer having a circular opening 1.75 in. in diameter is clamped on the lower end of the mold and the load then applied at a rate which will cause the testing head to be de-

pressed 1 in. in 25 seconds. As the pressure increases, the briquette will be forced through the orifice of the circular washer. The reading is observed on a spring dynamometer and the maximum is recorded. The average of two specimens is taken as a measure of the "stability" of the mixture. This test indicates the resistance of the pavement to displacement and may be used to proportion

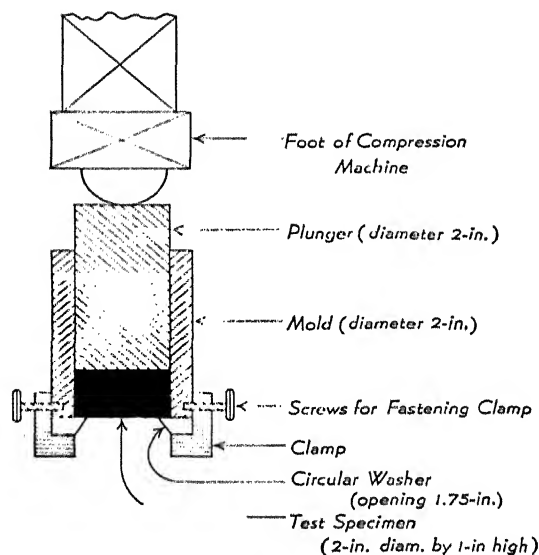


FIG. 340.—Apparatus for Testing Resistance to Displacement.

mixtures of sand, filler and asphaltic cement giving optimum results.

For testing coarse-aggregate paving mixtures¹⁵ a larger mold is recommended in conjunction with a testing ring $\frac{3}{8}$ in. thick, having a minimum orifice $5\frac{3}{4}$ in. in diameter for a depth of $\frac{1}{8}$ in., after which it tapers out to a diameter of 6 in. at the bottom of the ring.

Samples of the finished mixture taken from the testing plant are warmed to 250–260° F. and formed into briquettes as described above. Finished pavements may be tested by cutting out cores 2 in. in diameter and approximately $1\frac{1}{4}$ in. high, which are then placed directly in the mold and tested at 140° F.

A modified procedure (for testing resistance to compression) has been proposed, involving testing a cylindrical specimen 4 in. diameter by 2½ in. high, in a machine illustrated in Fig. 341. The specimen at 60° C. is surrounded by a rubber sleeve forming a watertight chamber with the metal casing, which is filled with water at 5 lbs. per sq. in. The pressure is increased at 0.05 in. per min.

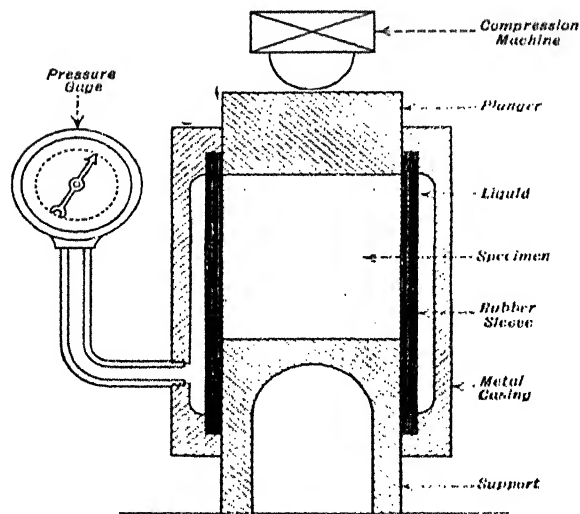


FIG. 341.—Testing Resistance to Compression.

until the test load of 400 lbs. per sq. in. (T) is developed, whereupon the gauge reading (G) is noted. The "stability" of the specimen is calculated from the formula: $\frac{100 (T-G)}{T}$. Values for road materials are in the range of 80.¹⁴

(II) *For Testing Soil Stabilized with Emulsified Asphalt.* The method has been modified as follows:¹⁵ Cylinders are formed 2 in. diameter by 4 in. high, at a moisture content which will produce dense specimens. These are dried at 140° F. and placed in the testing cylinder (Fig. 342). A load of 3,000 lbs. is applied while the bottom orifice is closed with a plug. The plug is then removed and a load applied until the plunger has been moved

downward $\frac{1}{2}$ in., thereby extruding a portion of the cylinder, and recorded in pounds.

Test 47a. Settling Tendencies of Fillers. The following procedure has been standardized: ^{15a}

This method of test is used as a measure of the rate at which mineral matter in mixtures of asphalt and mineral matter will settle when maintained without agitation at a temperature of 325° F.

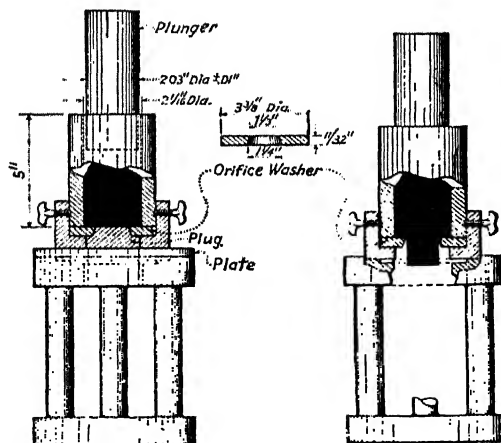


FIG. 342.—Stability Test Apparatus for Stabilized Soil.

The following apparatus is required:

(a) A special cylindrical container with walls of 4 in. inside diameter, standard pipe or seamless steel tubing with $\frac{1}{4}$ -in. wall and provided with two drainage taps with openings centered on vertical centerline of the shell $\frac{3}{4}$ in. and 2 in. above the bottom of the cylinder. Capacity approximately 70 cu. in. (See Fig. 342A.)

(b) Laboratory oven of sufficient size to admit special cylinder above, with temperature control to $325 \pm 1^{\circ}$ F. ($163 \pm 0.6^{\circ}$ C.).

The sample shall consist of at least 2 qt. of the asphalt mineral filler which shall be warmed to liquid condition and thoroughly stirred to insure uniform composition.

Procedure: After vigorous stirring take a 5-g. sample of the asphalt mineral filler mixture for determination of inorganic matter (ash) in accordance with provisions of Standard Method of

Test for Inorganic Matter or Ash. The sample shall then be transferred to the warmed special settlement container filling it to within $\frac{1}{4}$ to $\frac{3}{16}$ in. from the top. The containers shall then be set in the oven maintained at $325 \pm 1^\circ \text{F.}$ ($163 \pm 0.6^\circ \text{C.}$) for 2 hrs. without agitation.

At the end of the 2-hr. period remove the container and place on level surface where the upper drainage tap can be opened and

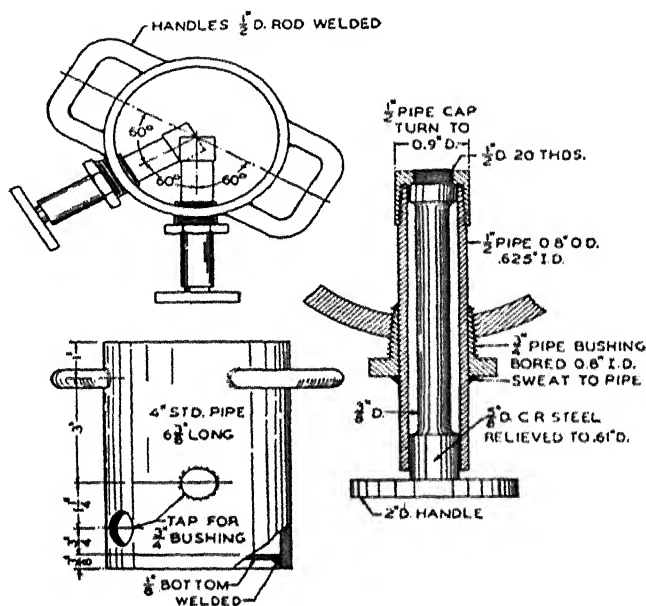


FIG. 342A.—Settlement Ratio Apparatus.

the asphalt above this tap removed. Next open the lower drainage tap and remove liquid to that level. When flow has practically ceased, tilt the container away from the bottom tap and remove both taps, replacing the bottom one with the pipe plug.

In removing the two taps do not dislodge the mineral matter which has settled on the top of the drainage tubes.

Next place the container over a gas flame or hot plate to make the remaining contents liquid. Thoroughly stir the remaining as-

phalt and uniformly incorporate all mineral matter remaining in the container.

Take another 5-g. sample for determination of inorganic matter (ash).

The percentage of ash found in the bottom sample remaining in the container, divided by the percentage of ash found in the sample before the settlement test, shall be reported as the settlement ratio.

Test 47*b*. **Shearing Strength.** This is ascertained in the 2-in. Hubbard-Field tester (Test 47), special clamps being provided in place of the extrusion die and mold. One end of a test briquette (19 in. long, by $2\frac{1}{4}$ in. wide, by $1\frac{1}{2}$ or 2 in. thick as required) is supported in a closely fitting socket, forming one clamp, and another U-shaped clamp is placed over the projecting end of the briquette against the socket, and held stationary, while the socket is forced upward at approx. $\frac{1}{25}$ in. per second, until the specimen shears. The maximum load sustained by the specimen divided by its cross-sectional area is taken as the shear strength.

Test 48. **Extrusion of Binder under Pressure.** A rapid test has been proposed¹⁶ for ascertaining by colorimetric means any excess of bituminous binder present in paving mixtures by means of a so-called "compression stain print." A briquette of the paving mixture, of cylindrical form, 1 to 2 in. high having a cross-sectional area of 3 sq. in., is prepared as in Test 47 and subjected to a pressure of 6000 lb. for 12 minutes at a temperature of 300–375° F., one surface being placed in contact with a sheet of kraft paper. The stain produced on the paper may be observed as an indication of the excess or deficiency of the bituminous binder present in the mixture. Thus the degree of discoloration or stain is proportionate to the relative amount of binder present, and an experienced observer will quickly learn to judge by eye whether there is an excess or deficiency of binder, and approximately the amount of such excess or deficiency.

Test 48*a*. **Flow Test.** Used for testing bituminous enamel as follows:¹⁷

Procedure: The plate prepared for observation of the workability of the enamel shall be used for this test. Lines 1 in. apart shall be drawn horizontally across the face of the enamel and to

the edge of the plate. The plate so prepared shall be suspended for five hours in a vertical position in an oven or heated chamber which is maintained at a constant temperature of 212° F. In order to secure uniformity of temperature and other conditions, the oven shall be equipped with a mechanically driven vertical shaft passed through the center of the top of the oven, from which shaft arms extend at right angles to it. On these cross-arms the coated steel plates shall be hung and the shaft revolved at 6 to 10 revolutions per minute. A thermometer shall be carried on one of the cross-arms so that the temperature of the thermometer will be the temperature to which the plates are subjected. At the end of five hours the plates shall be removed and during this test the coating must not have slipped in any part more than one-fourth inch as indicated by the lines drawn across the coating and plate.

Test 49. Resistance to Impact. This test was originally devised by L. W. Page for testing the toughness of rock for road building,¹⁸ having since been adapted by Clifford Richardson for testing bituminous aggregates.¹⁹ The bituminous mixture is heated to the lowest possible temperature that will permit its being manipulated, and formed by compression into a cylinder 25 mm. high by 24–25 mm. in diameter, the ends of which shall be plane surfaces at right angles to its axis. The hot bituminous mixture is compressed in a hollow cylindrical steel mold, 24–25 mm. in diameter by 50 mm. long, having an accurately fitting steel plunger. The mold is loosely filled with the hot bituminous mixture and compressed with the plunger by sharp blows of a heavy hammer from the top and bottom respectively, until it is thoroughly compacted. The cylinder of bituminous material is then knocked from the mold and sawed off or ground down until it measures exactly 25 mm. high. The density of the specimen should be noted and reported. It shall be maintained in water at 77° F. for 48 hours, wiped dry, and tested in air at a temperature of 77° F. on any form of impact machine which will comply with the following essentials:

(a) A cast-iron anvil weighing not less than 50 kg. firmly fixed upon a solid foundation.

(b) A hammer weighing 2 kg. arranged to fall freely between suitable guides.

(c) A plunger of hardened steel weighing 1 kg. arranged to slide freely in a vertical direction in a sleeve, the lower end of the plunger being spherical, with a radius of exactly 1 cm.

(d) Means for raising the hammer and dropping it upon the plunger from any specified height from 1 to not less than 75 cm.

(e) Means for holding the cylindrical test-specimen securely on the anvil without rigid lateral support, and under the plunger in such a way that the center of its upper surface shall, throughout the test, be tangent to the spherical end of the plunger at its lowest point.

The test shall consist of a 1-cm. fall of the hammer for the first blow; a 2-cm. fall for the second blow; and an increase of 1 cm. for each succeeding blow, until failure of the test specimen occurs. The number of blows required to shatter the test-piece is taken to represent the toughness, three such tests being averaged. Tests are performed at three temperatures, viz.: 32° F., 77° F. and 115° F.

Another method has been proposed for testing pavements,²⁰ in which specimens of full thickness are constructed as slabs 3 ft. square, upon a rolled cinder subgrade, and tested in place by allowing a 125-lb. iron ball to drop from a height of 6 in. at the rate of 30 blows per minute for the first 1000 blows, 7 in. for the next 200 blows, and an increase of 1 in. for each successive 200 blows until the first crack appears, which is regarded as the failure of the specimen. Fig. 343 shows the behavior of various types of pavements under this test. Concrete specimens were cured under moist sand and tested when twenty-eight days old.

Test 50. Brittleness or Shatter Test. Used for testing bituminous enamel as follows:²¹

This test shall be made on the sample which has been used for the workability test, and shall be conducted as follows: A 750-g. (1.65-lb.) steel ball shall be dropped from a height of 244 cm. (8 ft.) on the coating at a point at least 10 cm. (4 in.) from the edge of the plate. The plate is to be supported in this test by a block or the floor in such a manner that there shall be support beneath the point of impact. In this test none of the coating shall be shattered from the plate nor shall radical cracks develop longer than 4 cm. (1.6 in.), from the center of the point of impact. The coated steel plate shall then be laid coating downward, on a board through which a hole about 8.9 cm. (3.5 in.) in diameter has been cut. The same steel ball shall be dropped from a height of 244 cm. (8 ft.) and shall strike the steel plate over the center of the hole in the board supporting the plate. The coating must not shatter from the plate by this test, nor shall cracks develop in the coating farther

from the center of point of impact than the boundaries of the hole in the board.

Test 51. Coefficient of Wear. This may be measured by a sandblast under carefully controlled conditions.²² Sand of predeter-

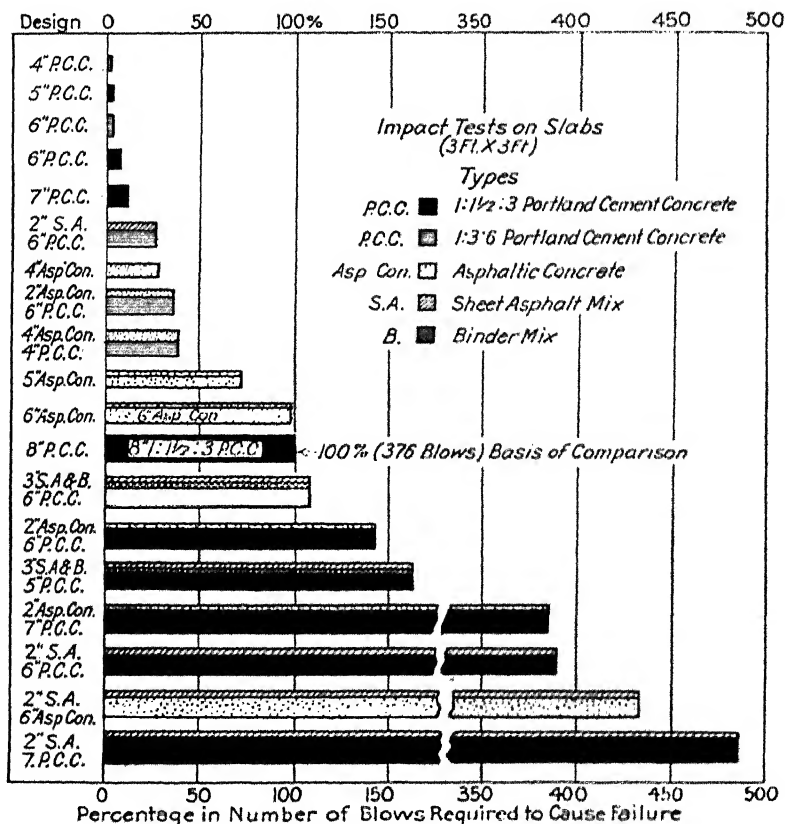


FIG. 343.—Resistance of Typical Pavements to Impact.

mined size is projected against an area 6 cm. in diameter, under a pressure of 3 atmospheres. For further details, the reader is referred to the article given in the reference.

Another test has been proposed²³ in which a section of the

pavement is laid in a 14-ft. diameter circular track and subjected to the action of a metal roller, or to a pneumatic tired wheel under accurately controlled conditions.

(2) MOLDED MATERIALS ²⁴

Test 51a. Thickness. This test has been standardized as follows: ²⁵

These methods cover procedures for determining the thickness of solid insulating materials, except rubber insulating tape and friction tape for electrical purposes. Three alternative procedures are described, as follows:

Method I makes use of an adjusted ratchet micrometer together with a definite manipulative procedure by which the pressure exerted on the specimen is controlled.

Method II, known as the "feel" method, makes use of a machinist's micrometer constructed without a ratchet, pressure on the specimen being controlled by stopping closure of the micrometer when resistance to movement of the specimen between the instrument surfaces is first observed.

Method III makes use of a dead weight dial micrometer, which is constructed so that measurements made with it are practically the same as those made with the Method I ratchet micrometer. Method I and Method III instruments may be used interchangeably.

Methods I and III are preferred as reference standard methods for use in cases of dispute. They are also preferred for the measurement of compressible materials such as untreated paper and fabrics, as well as for the measurement of rigid materials. The maximum error (instrumental plus manipulative) of Methods I and III is of the order of 0.0003 in.

Method II may be used on rigid materials or on yielding materials where it is necessary to measure the specimen with practically no compression or deformation. The maximum error of Method II is approximately 0.0005 in., except on unusually compressible materials, where the error will be somewhat greater.

Method I

The instrument used for determining thickness by Method I shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with a vernier reading to 0.1 mil (0.0001 in.) and with a ratchet or similar mechanism for controlling measured pressure, and shall have anvil and spindle surfaces 0.250 in. \pm 0.001 in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, wear of micrometer screw, micrometer screw error tolerances and ratchet pressure specified below. The micrometer shall be tested and calibrated periodically for conformity to these requirements.

Before starting measurements of thickness the micrometer shall be closed on the specimen at a location outside the area to be measured. The micrometer shall then be opened not more than 4 or 5 mils (0.004 or 0.005 in.) and then moved into the area selected for measurement. Using the ratchet, the micrometer surfaces shall be closed so slowly on the specimen that the mil scale divisions may be easily counted as they move past the reference mark, or at a rate of about 2 mils (0.002 in.) per second. The closing motion shall be continued at the same rate until the ratchet has clicked three times, and then the thickness shall be read by means of the vernier.

In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or 0.005 in.) more than the specimen thickness. In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least $\frac{1}{4}$ in. from the edges of the specimen.

Method II

The instrument used for determining the thickness by Method II shall be a 1-in. machinist's type micrometer without a locking device. It shall be constructed with vernier reading to 0.1 mil (0.0001 in.) and with anvil and spindle surfaces 0.250 in. \pm 0.001 in. in diameter. The instrument shall conform to the requirements for flatness and parallelism of micrometer surfaces, zero reading, and micrometer screw error tolerances specified below. The mi-

chrometer shall be tested and calibrated periodically for conformity to these requirements.

In the determination of thickness, the micrometer specified shall be slowly closed on the specimen until contact is made, without appreciable distortion of the specimen. The criterion of contact is the initial development of frictional resistance to movement of the specimen between the micrometer surfaces. The thickness shall then be read by means of the vernier.

In moving from one measurement location to another this operation shall be repeated, never opening the micrometer more than 4 or 5 mils (0.004 or 0.005 in.) more than the specimen thickness.

(a) The micrometer shall be a dead weight dial type micrometer, having two ground and lapped circular surfaces, each 0.250 ± 0.001 in. in diameter, and shall have a capacity of not less than 0.170 in.

(b) The surfaces shall be parallel to within 0.0001 in. and shall move on an axis perpendicular to themselves.

(c) The pressure exerted on the specimen shall be within the limits of 23 and 27 lb. per sq. in.

(d) The dial spindle shall be vertical and the dial shall be at least 2 in. in diameter. It shall be continuously graduated to read directly to 0.0001 in. and shall be equipped with a telltale hand, recording the number of complete revolutions of the large hand. The dial indicator mechanism shall be full jeweled.

(e) The micrometer shall be capable of repeating its readings to 0.00005 in. at zero setting or on a steel gage block.

(f) Measurements made on standard steel gages shall be within the following tolerances:

Intervals, in.	Permissible Deviation of Reading from Actual Thickness of Standard Steel Gage, in.
0 to 0.01	0.0001
Over 0.01	0.0005

(g) The deviations for the parts of the scale corresponding to the paper thickness measured, shall be applied as corrections to the thickness reading.

(h) The frame of the micrometer shall be so rigid that a load of 3 lb. applied to the dial housing, out of contact with either the weight or the presser foot spindle, will produce a deflection of the frame not greater than 0.0001 in., as indicated on the micrometer dial.

The micrometer shall be placed on a solid, level table, free from excessive vibration. The specimen shall be placed between the micrometer surfaces and the presser foot lowered on the specimen at a location outside the area to be measured. The presser foot shall then be raised a distance of 0.3 to 4.0 mils, the specimen moved to the measurement position and the presser foot dropped onto the specimen.

NOTE.—The procedure described minimizes small errors present when the presser foot is lowered slowly on the specimen. Care should be taken not to raise the presser foot more than 0.4 mil above the position of rest on the specimen surface.

In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least $\frac{1}{4}$ in. from the edges of the specimen.

For each succeeding measurement, the presser foot shall be raised 0.3 to 0.4 mils, the specimen moved to the next measurement location and the presser foot dropped.

When compressible papers or fabrics are measured, a slight settling of the presser foot occurs. To minimize the errors produced by this effect, the reading of the dial indication should be deferred until the presser foot has been supported by the specimen for at least 2 sec., or until the micrometer hand becomes stationary.

Before and during instrument calibration and thickness measurements, the micrometer surfaces shall be maintained in a clean condition by lightly closing them on a clean sheet of bond paper and moving the paper between the surfaces. To minimize the danger of the presence of lint, the edge of the paper should not be pulled between the surfaces.

"I" Micrometer.—In the calibration of controlled pressure micrometers used in Method I, the micrometer shall be closed on the gage or calibrating device and then opened 4 or 5 mils (0.004 or 0.005 in.). Using the ratchet, the micrometer shall be again closed so slowly on the calibrating device that the mil scale divisions may be easily counted as they move past the reference mark, or at the rate of about 2 mils (0.002 in.) per sec. The closing motion shall be continued at the same rate until the ratchet has clicked three times, when the reading shall be taken.

"II" Micrometer.—In the calibration of micrometers used in Method II the micrometer shall be slowly closed on the gage or

calibrating device until contact of the surfaces and gage is made. The criterion of contact is the initial development of frictional resistance to movement of the gage device between the micrometer surfaces.

In making a measurement, all points on the peripheries of the micrometer surfaces shall be at least $\frac{1}{4}$ in. from the edges of the specimen.

The anvil and spindle surfaces of the micrometer shall be flat to within 0.00005 in. The flatness may be determined by use of an optical flat, as follows: After cleaning the surfaces of the flat and the micrometer the latter shall be closed on the flat as described above. When illuminated by diffused daylight, interference bands are formed between the surfaces of the flat and those of the micrometer. The location, shape and number of these bands indicate the deviation from flatness in increments of half the average of the wave lengths of white light, which is taken as 0.00001 in.

A *flat surface* forms straight, parallel and equidistant fringes.

A *grooved surface* forms straight parallel fringes at unequal intervals. The estimated maximum displacement of any line from its normal position, where all lines would be equidistant, is a measure of deviation from flatness.

A *symmetrical concave or convex surface* forms concentric circular fringes, and their number is a measure of deviation from flatness.

An *unsymmetrical concave or convex surface* forms a series of curved fringes, cutting the periphery of the micrometer surface. The number of fringes cut by a straight line connecting the termini of any fringe is a measure of the deviation from flatness.

The anvil and spindle surfaces of the micrometer shall be parallel to each other to within 0.0001 in. when tested with a pair of screw-thread pitch wires or with a pair of $\frac{1}{4}$ -in. nominal diameter plug gages. The diameters of the screw-thread-pitch wires or the plug gages, accurate to 0.00002 in., shall differ by an amount approximately equal to the axial movement of the spindle when rotated through 180 deg. (12.5 mils). The micrometer shall be closed on the wires or on the plug gages according to the procedure described above. Observations, made with either wire or with either plug gage placed at any location between the surfaces, shall

show differences of less than 0.0001 in. The position of the anvil shall be such that a zero reading is obtained when the micrometer is closed on the anvil as described above. Ten trials shall give ten readings of zero.

The condition of zero reading is satisfied when examinations with a low-power magnifying glass show that at least two-thirds of the widths of the zero graduation on the barrel and that of the reference mark coincide with each other.

The device for compensating for wear of the micrometer screw shall be adjusted so that the spindle has no perceptible lateral or longitudinal looseness and yet may be rotated with a torque load of not more than $\frac{1}{4}$ in.-oz.

The micrometer screw error, after zero adjustment is made, shall be checked at 2, 5, and 10 mils, and at intervals of 100 mils over the remaining graduated scale. For checks up to and including a thickness of 10 mils, selected gage blades the thicknesses of which are known to ± 0.00002 in. shall be used. Checks at values greater than 10 mils shall be made with standard gage blocks. At each value checked, ten readings shall not differ from the thickness of the gage used by more than 0.1 mil (0.0001 in.). Manipulation of the instrument in these checks shall be in accordance with the above. The ratchet shall be so adjusted that a pressure of not more than 27 nor less than 23 lb. per sq. in. is developed when the spindle surface is contacted with a polished steel surface as described.

In making these calibration measurements, the presser foot shall be raised from 0.3 to 0.4 mil above the position of contact with the steel ball or gage. After dropping the presser foot from the elevated position, the thickness reading shall be observed.

Parallelism of Surfaces: A hardened steel ball about $\frac{1}{16}$ in. in diameter, fixed firmly in a flat metal handle about $\frac{1}{32}$ in. in thickness, shall be measured at several locations on the micrometer surfaces, and the maximum variations of readings noted.

Accuracy of Scale Divisions: The instrument shall be set at zero and standard gages, the thickness of which is known to within 0.00001 in., shall be measured.

Pressure: The pressure applied to the presser foot spindle and weight necessary to move the pointer upward from the zero posi-

tion shall not be greater than 650 g. The pressure applied to the presser foot spindle and weight necessary to just prevent movement of the pointer from a positive toward a lower reading shall not be less than 500 g.

Test 51b. Expansion in Boiling Water. The following procedure has been proposed for testing preformed bituminous expansion joints to determine the extent of expansion in boiling water: ²⁶

Five test specimens measuring $4\frac{1}{2}$ by $4\frac{1}{2} \pm 0.10$ in. shall be cut from each sample. The thickness of each test specimen shall be determined to the nearest 0.001 in. The specimens shall be immersed in boiling water for 1 hour, after which they shall be removed and allowed to cool to room temperature for 15 minutes. The final thickness of each specimen shall then be measured to the nearest 0.001 in.

Test 52. Resistance to Moisture. The following method has been standardized: ²⁷ The test specimen shall be molded in the form of a disk 4 in. (10.16 cm.) in diameter, which should be $\frac{1}{8}$ in. (3.18 mm.) in thickness for hot-molded materials, and $\frac{1}{4}$ in. (6.35 mm.) in thickness for cold-molded materials.

NOTE.—To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds.....	± 5 per cent
For cold-molded compounds.....	± 10 per cent

Three specimens shall be tested. Each of the three shall be weighed separately in the as-received condition. If the material softens readily at moderate temperatures, the specimen may be placed in a desiccator for twenty-four hours, or in an oven at a temperature of 50° C. (122° F.), permissible variation $\pm 5^\circ$ C., for twenty-four hours. For materials which do not soften readily, the specimen shall be placed in an oven heated to 100° C. (212° F.), permissible variation $\pm 5^\circ$ C., for twenty-four hours. After drying, the pieces shall be cooled in a desiccator and weighed again at normal room temperature. The specimens shall be placed in water, wholly immersed, for forty-eight hours at a temperature of 25° C. $\pm 2^\circ$ C. They shall then be removed from the water at the end of forty-eight hours, all surface water wiped off with a dry cloth, and the specimens weighed immediately.

The report shall include the following:

- (a) The original weight of each specimen;
- (b) The dry weight of each specimen;
- (c) The weight of each specimen after immersion for forty-eight hours;
- (d) The percentage of water contained in each test specimen as received, and the percentage of moisture absorbed during the forty-eight hours, taking the dry weight as 100 per cent.

All weights shall be given in grams.

The following alternate procedure has been proposed²⁸ for testing sheet and plate materials intended for electrical insulation:

The water absorption tests are intended to determine the rate at which water is absorbed by the material when immersed and the total quantity absorbed at saturation.

NOTE.—The electrical properties of different materials are not necessarily affected to the same extent by the same increase in moisture content, so that the water absorption test must be correlated with the desired electrical tests.

The water immersion test and exposure to air of high humidity are not always directly comparable and should be considered before substituting one for the other.

Rate of Absorption: The rate of water absorption of all sheet and plate insulating materials shall be determined in accordance with A.S.T.M. Designation: D 570.

Total Absorption at Saturation: The test specimen shall be 3 by 1 in. by the thickness of the material for materials $\frac{1}{8}$ in. in thickness or less. For materials over $\frac{1}{8}$ in. in thickness the specimen shall be 3 by $\frac{1}{8}$ in. by the thickness of the material. It shall be sawed or sheared from the sample so as to have smooth edges free from cracks. The cut edges of all test specimens shall be finished with No. 0 or finer sandpaper or emery cloth. The sawing and sandpapering operations should be slow enough so that the material is not heated appreciably. The thickness in inches to the nearest 0.001 in. shall be measured in the direction perpendicular to the faces of the original sample.

To determine the total water absorbed at saturation, three specimens shall be tested individually, weighed, and then conditioned as described below.

Materials whose water absorption value would be appreciably affected by temperature in the neighborhood of 110° C. shall be dried in an oven for 24 hours at $50 \pm 3^\circ$ C., cooled in a desiccator, and immediately reweighed.

Specimens of materials such as phenolic laminated, and other products whose water absorption value has been shown not to be appreciably affected by temperatures up to 110°C . shall be dried in an oven for 1 hour at 105 to 110°C .

The dried specimen shall be immersed in distilled water at 20 to 30°C . At the end of 24 hours the specimen shall be removed from the water, the surface water wiped off with a dry cloth, and the specimen weighed immediately and then replaced in the water. The weighings shall be repeated every day for the first week and every week thereafter until the increase in weight, as shown by three consecutive weighings, averages less than 1 per cent of the total increase in weight, when the specimen shall be considered to be saturated.

The difference between the saturated weight and the dry weight shall be considered as the water absorbed at saturation.

The report shall include the test results for each of the three specimens as follows:

- (a) The original thickness of material measured to the nearest 0.001 in.,
- (b) The percentage loss in weight on drying, calculated on the original weight, and
- (c) The percentage of water absorbed at saturation, calculated on the dry weight.

Tests have also been standardized for measuring the shrinkage on molding.²⁰

Test 53. Tensile Strength. This has been standardized as follows:²⁰

Any universal testing machine may be used, provided it is accurate to 1 per cent of the lowest load to be applied. Special specimen holders made of steel, as shown in Fig. 344 (a), shall be used for such test specimens as are of a shape to fit them. The specimen holders shall be attached to the heads of the testing machine by links held so that the pull is central to avoid any transverse strain. Equally suitable holders, suitably mounted, shall be used for specimens of other shapes.

For hot-molded materials and for plastic cold-molded materials (Note 1), the test specimen shall be molded to the form and dimensions shown in Fig. 344 (b), the use of either the $\frac{1}{8}$ -in. or the $\frac{1}{4}$ -in.

specimen being permissible (Note 2). Five specimens are required for a test. Special specimen holders made of steel, as shown in Fig. 344(a), shall be used.

NOTE 1.—Due to wide differences in molding characteristics, the specimen suited to hot-molded and plastic cold-molded materials is unsuited to the nonplastic inorganic cold-molded materials.

NOTE 2.—Values of tensile strength obtained with these two specimens may be unequal and accordingly that specimen should be chosen which is considered most representative of the material and its intended use.

Five conditioned specimens shall be tested for each type of molded material, each specimen being properly aligned in the holders and broken. The crosshead speed of the testing machine shall be such that the load can be accurately weighed but shall not exceed 0.050 in. per min. (1.27 mm. per min.) with the machine running idle. All tests shall be made at normal room temperature of about 20° C. (68° F.).

The report shall include the following:

(a) The breaking load of each specimen in pounds or kilograms;

(b) The thickness and width of each specimen in inches or millimeters as measured by a micrometer at the center of the specimen, that is, the point of minimum section;

(c) The ultimate tensile strength in pounds per square inch or in kilograms per square millimeter of each specimen, calculated from the minimum area measured at the center of the test specimen before the load is applied;

(d) The character of the material tested, with description of the fracture and its location with respect to the middle point of the specimen.

(e) The speed in inches or millimeters per minute at which the head of the testing machine traveled during the test.

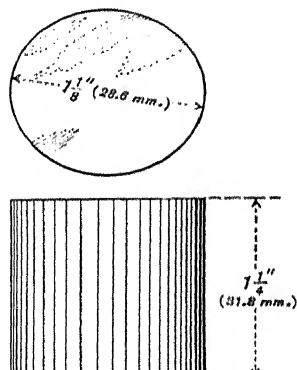
Various modifications in tensile-strength procedures have been proposed from time to time.⁸¹

Test 53a. Compressive Strength. (I) *For Testing Molded Compositions:* This method has been standardized as follows:⁸²

Any universal testing machine may be used provided it is accurate to within 1 per cent of the lowest load for which it is used. One end of the specimen shall bear upon an accurately centered spherical bearing block, located whenever practicable at the top,

and the metal bearing plates shall be directly in contact with the ends of the test specimen.

Test specimens shall be of the size prescribed for the material being tested. A primary requirement for all test specimens is that the ends shall be flat and perpendicular to the axis. Unless more particularly specified elsewhere the dimensions should conform to the following:



Courtesy A.S.T.M.

FIG. 345.—Compression Test Specimen.

Molded Materials: A cylinder as illustrated in Fig. 345, having a height of 1.25 in. (31.8 mm.) and a diameter of 1.125 in. (28.6 mm.).

Laminated Tubes: A section of the tube 1 in. in length.

Sheet and Plate: A 1-in. cube for sheets 1 in. or over in thickness; for sheets less than 1 in. in thickness a pile of sheets 1-in. square with a minimum number of layers to produce a height of at least 1 in.

Round Rods: A section of the rod.

NOTE.—To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds . . .	± 5 per cent
For cold-molded compounds . . .	± 10 per cent

The design of the mold and molding conditions to be used to obtain concordant results shall be agreed upon by the purchaser and the seller. Due to the size and shape of the compression test specimen, it is sometimes difficult to cure it completely within the relatively short time which generally would be used, and with the temperatures ordinarily employed. This is particularly true when hand molds, that is, molds heated by conduction from the press platens, are used. It is, therefore, recommended that special precautions be taken to insure that the specimen is thoroughly cured, experiments indicating that 20 to 30 minutes may be necessary in some cases with hand molds.

Five conditioned specimens shall be tested at room temperature, or at other temperatures as specified.

The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. (1.27 mm. per min.) when the machine is running idle.

Molded materials shall be tested with the load applied on the ends of the specimen.

Laminated tubes shall be tested axially with the load applied perpendicular to the faces or ends of the specimen. Specimens shall also be tested diametrically with the load applied perpendicular to the tangent at the point of application.

Rods shall be tested with the load applied perpendicular to the faces or ends of the specimen.

Sheet and plate materials 1 in. and over in thickness shall be tested both flatwise and edgewise, and cut both crosswise and lengthwise of the sheet. Thinner sheets shall be tested only flatwise and in piles.

The report shall include the following:

(a) The significant dimensions of each specimen in inches measured to the nearest 0.001 in., or in millimeters to the nearest 0.025 mm. and the direction of cutting for sheet and plate specimens. Thickness is the thickness of sheet or plate, or the dimension parallel to the application of molding pressure for molded specimens,

(b) The load on each specimen in pounds or kilograms at the first sign of failure and the direction of application of the load,

(c) The ultimate compressive strength in pounds per square inch, or kilograms per square millimeter, for molded materials and sheets and plates calculated from the measured area before the load is applied and for rods and tubes calculated from the data obtained on the application of the load perpendicular to the face of the specimen,

(d) The rate at which the load was applied, and

(e) Any condition of the specimen or test which has not been standardized for the material.

Additional tests may be made at elevated temperatures, the actual temperatures selected depending upon the use that is to be made of the material. Five specimens shall be tested at each temperature selected. The specimen shall be kept at that temperature a sufficient length of time to become of uniform temperature throughout and shall be maintained at that temperature during the test.

NOTE 1.—A convenient method of making this test is to submerge the specimens in a suitably lagged, electrically-heated oil bath of about two gallons capacity. The specimens should be placed in the bath at least 30 minutes before testing and the temperature maintained approximately constant at the desired testing temperature until the completion of that test. A pale mineral oil having a viscosity of 100 seconds ± 10 seconds at 38° C. (100° F.) Saybolt is satisfactory for this purpose. The specimens should not be placed in the oil an excessive time in advance of testing. The oil bath should be well stirred to insure uniform temperature throughout. The temperature should be determined by means of a thermocouple sealed in a similar specimen in the bath; or by the use of an A.S.T.M. partial-immersion thermometer conforming to the Standard Specifications, so inserted in a hole in a specimen that the bulb is completely enclosed and sealed in.

If tests are required over the whole range of temperature rather than at particular points, the method of testing may be found simpler and quicker if modified in accordance with Note 2, below.

NOTE 2.—The oil with 5 specimens immersed in it should be heated to the highest temperature to be used and allowed to cool slowly. Specimens should be tested in succession and the average temperature during the actual time of testing of each specimen taken as that corresponding to each strength determination. Specimens should be added to the bath successively on such a schedule that each will remain in the bath at least 30 minutes before testing. An average curve should be drawn through the plotted data.

In certain cases mixtures of bituminous constituents with mineral fillers may be prepared at 325° F., and when the temperature falls to 305–315° F., compressed in a mold heated to 310° F. under a load of 5000 lb. per sq. in. maintained for 10 seconds. When coarse mineral matter is used, the dimensions of the mold may be suitably increased (e.g., 2 in. in diameter by 1 in. high, or larger). In the case of a finished product, specimens may be conveniently cut by means of a carborundum saw. The various factors constituting the shearing resistance of bituminized aggregates have been investigated from the theoretical viewpoint.²²

(II) *For Testing Bituminous Expansion-joints:* The following procedure has been standardized for ascertaining the extent of compression and recovery of bituminous expansion-joints used for concrete pavements:²⁴

Mounting: The test specimen shall be placed on a flat metal plate, and a 4½ by 4½ by ½-in. metal plate ground to have plane parallel faces shall be centered on the top surface of the specimen. A simple U-shaped bridge shall be employed to support a dial or other suitable measuring device reading to 0.001 in. above the center of the specimen. A metal cylinder or other device for transferring the load from the moving head of the testing machine around the measuring apparatus to the plate covering the speci-

men shall be placed upon the plate. A spherical bearing block shall be mounted between the upper end of the cylinder and the moving head of the testing machine.

Measurement of Thickness: When the specimen has been mounted as described and is subjected only to the pressure of the dead weight of the $4\frac{1}{2}$ by $4\frac{1}{2}$ by $\frac{1}{2}$ -in. metal plate, its thickness shall be determined by means of the measuring device. When the load transferring apparatus and spherical bearing block are placed on the test specimen, some compression may result. This reduction in thickness shall be considered as part of the 50 per cent reduction in thickness to be applied.

Recovery: For the determination of the percentage of recovery, the test specimen shall be given three applications of a load sufficient to compress it to 50 per cent of its thickness before test. The load shall be applied without shock and at such a rate that the specimen will be compressed approximately 0.05 in. per min. After the first and second applications, the load shall be released immediately, and the specimen permitted to recover 30 min. before the load is again applied. After the third application, the load shall be released immediately and the specimen shall be permitted to recover 1 hr., after which the thickness shall again be measured. The load-transferring apparatus and spherical bearing block shall be removed from the test specimen during recovery periods between compressions and following the third application of load. The percentage of recovery shall be calculated as follows:

$$\text{Recovery, per cent} = \frac{t'}{t} \times 100$$

where t = thickness of the specimen before test, and

t' = thickness 1 hour after completion of the third application of load.

Compression: The total maximum load in pounds required for the first application as specified shall be divided by 16 and recorded as the unit pressure in pounds per square inch.

Test 54. Flexural Strength. This test has been standardized as follows: ²⁵

Any universal testing machine may be used provided it is accurate to 1 per cent of the lowest load to be applied. The specimen

shall be tested as a simple beam, resting on two parallel supports, and loaded at the center by means of a pressure piece to apply the load crosswise of the beam. The distance between supports shall be eight times the nominal height of the specimen in test position but not less than $\frac{1}{2}$ in., or for laminated round rods not more than 4 in. Pins may be added to the supports to keep very narrow specimens in a vertical position. The radius of all contact edges shall be $\frac{1}{8}$ in. (3.18 mm.), except as otherwise specified.

Test specimens shall be of the size specified for the material being tested, but unless more particularly specified elsewhere, should conform to the following:

Molded Materials: Specimens $\frac{1}{2}$ by $\frac{1}{2}$ by 5 in. or of such lesser thickness as is considered representative as illustrated in Fig. 346.



FIG. 346.—Test-specimen for Flexural Strength.

Sheets and Plates: Width of $\frac{1}{2}$ in., except for specimens over $\frac{1}{2}$ in. in thickness tested in the flatwise direction in which case the width shall be equal to the thickness of the specimen. The thickness shall be the full thickness of the sheet. The length shall be 1 in. greater than the distance between supports.

Laminated Round Rods: A section of rod 5 in. in length.

The thickness of the molded specimen, that is, the dimension parallel to the application of the molding pressure may be any value of $\frac{1}{2}$ in. or less agreed upon as representative of the cross-section in which the material is to be used. The thickness as molded will be the width of the beam when under test (Note). Five specimens are required for a test.

NOTE.—When the specimen is less than $\frac{1}{4}$ in. in molded thickness, difficulties may be encountered due to tipping over or unsteadiness during test. In such cases the test may be made upon a composite specimen consisting of sufficient individual pieces to aggregate approximately $\frac{1}{2}$ in. in beam width held together by a rubber band or other light clamp.

To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds..... ± 5 per cent
For cold-molded compounds..... ± 10 per cent

Not less than five specimens (conditioned when necessary) shall be tested at room temperature, or at other temperatures as specified, for each strength value to be determined.

The crosshead speed of the testing machine shall be such that the load can be accurately weighed, but shall not exceed 0.050 in. per min. (1.27 mm. per min.) when the machine is running idle. Measurements of deflection may be made for very elastic materials.

Molded materials shall be tested with the load applied crosswise of the specimen turned so that its thickness, or dimension parallel to the application of molding pressure, becomes the width of the beam.

Sheet and plate materials shall be tested with specimens turned both flatwise and edgewise, and cut both crosswise and lengthwise of the sheet.

The maximum fiber stress in pounds per square inch or kilograms per square centimeter shall be calculated as follows:

For bars of rectangular cross-section:

$$S = \frac{3Pl}{2bd^2}$$

For bars of circular section:

$$S = \frac{8Pl}{\pi d^3}$$

where S = maximum fiber stress,

P = breaking load in pounds or kilograms,

l = distance between supports in inches or centimeters,

b = width of the beam as tested in inches or centimeters, and

d = depth of the beam as tested or diameter of specimen in inches or centimeters.

The report shall include the following:

(a) The depth and width (or diameter determined from at least two measurements 90 deg. apart) of each specimen measured by a micrometer expressed in inches to the nearest 0.001 in. or in centimeters to the nearest 0.025 mm.,

(b) The direction of cutting and loading specimens of sheet and plate materials.

(c) The breaking load in pounds or kilograms,

(d) The maximum fiber stress in pounds per square inch or kilograms per square centimeter calculated as described above.

- (e) The rate at which the load was applied,
- (f) The maximum deflection at the center, in inches or millimeters if measured,
- (g) A description of the fracture, if significant,
- (h) Any condition of the specimen which has not been standardized for the material, and
- (i) Any other information required under a specific material test method.

Test 54a. Extrusion Test. The following procedure has been standardized for testing preformed expansion-joints: ¹⁰⁰

The test specimen shall be placed in a suitable steel mold so constructed as to confine the lateral movement of the specimen under compression to one side only. Interior dimensions of the mold shall be 4 by 4 in. with permissible variations in length and in width of plus or minus 0.015 in. Mold sides shall be of such height as to extend at least $\frac{1}{2}$ in. above the test specimen. The specimen shall then be covered with a $\frac{1}{2}$ -in. metal plate ground to have plane parallel faces. The metal plate shall be machined to fit within the three restraining sides of the steel mold snugly but without binding. A simple U-shaped bridge shall be employed to support above the center of the specimen a dial or other suitable measuring device reading to 0.001 in. A metal cylinder or other device for transferring the load from the moving head of the testing machine around the measuring apparatus to the plate covering the specimen shall be placed upon the plate. A spherical bearing block shall be mounted between the upper end of the cylinder and the moving head of the testing machine.

Measurement of Thickness: When the specimen has been mounted as described, and is subjected only to the pressure of the dead weight of the 4 by 4 by $\frac{1}{2}$ in. metal plate, its thickness shall be determined by means of the measuring device. When the load transferring apparatus and spherical bearing block are placed on the test specimen, some compression may result. This reduction in thickness shall be considered as part of the 50 per cent reduction in thickness to be applied.

Extrusion: For the determination of the amount of extrusion, the specimen shall be given one application of a load sufficient to compress it to 50 per cent of its thickness before test. The load

shall be applied without shock at such a rate that the specimen will be compressed approximately 0.05 in. per min. The amount of extrusion in inches shall be determined by measuring the maximum movement of the free edge of the test specimen during the 50 per cent compression of the specimen. The extrusion shall be measured by means of a dial or other suitable device reading to 0.001 in.

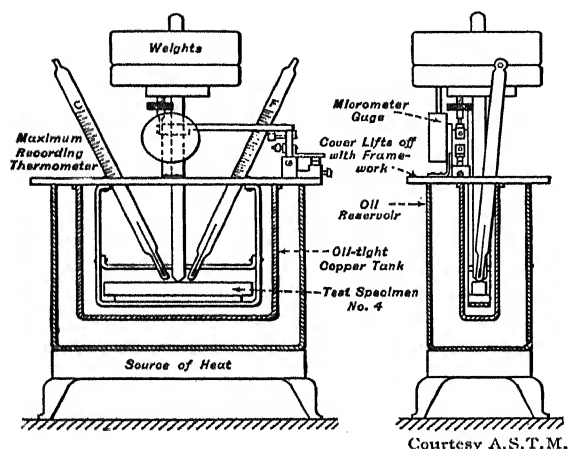


FIG. 347.—Machine for Distortion under Heat.

Test 55. Distortion under Heat. The following method has been standardized:³⁷

This method of test covers a procedure for determining the distortion under heat of all solid electrical insulating materials (except dry-process porcelain) that are formed in molds by the application of pressure, either with or without heat.

NOTE.—The type of mold used to produce test specimens has an effect on the results obtained. Cooperating laboratories should, therefore, standardize mold and testing procedure to obtain concordant results.

The apparatus shown in Fig. 347 shall be used. The specimen shall be supported on steel supports, 4 in. (102 mm.) apart with the load applied on top of the specimen vertically and midway between the supports. The contact edges of the supports and of the piece by which pressure is applied shall be rounded to a radius of $\frac{1}{8}$ in. (3.18 mm.). The machine shall be arranged to apply a

load of 5.5 lb. (2.5 kg.). The specimen shall be placed in an air bath surrounded by an oil bath so arranged that its temperature can be raised gradually. The machine shall be arranged so that the deflection of the specimen at its center between the supports can be measured on a scale in mils or millimeters and shall be equipped with a thermometer so that the temperature of the specimen can be recorded at any time. The machine may be arranged to shut off the heat automatically and sound an alarm as soon as the required deflection is reached.

Test specimens shall be of the size specified for the material being tested. In the absence of specific requirements, the specimens may be rectangular bars $\frac{1}{2}$ by $\frac{1}{2}$ by 5 in., with the thickness as molded as the width of the beam when under test.

The thickness of the molded specimen, that is, the dimension parallel to the application of the molding pressure, may be any value of $\frac{1}{2}$ in. or less, agreed upon as representative of the cross-section in which the material is to be used. The thickness as molded will be the width of the beam when under test (Note). Three specimens are required for a test.

NOTE.—When the specimen is less than $\frac{1}{4}$ in. in molded thickness, difficulties may be encountered due to tipping over or unsteadiness during test. In such cases the test may be made upon a composite specimen consisting of sufficient individual pieces to aggregate approximately $\frac{1}{2}$ in. in beam width held together by a rubber band or other light clamp.

To permit the use of one mold for all molding compounds the following variations in dimensions of the test specimen will be permissible:

For hot-molded compounds	± 5 per cent
For cold-molded compounds	± 10 per cent

Three test specimens shall be tested in the condition in which they are received, starting at a temperature $25 \pm 2^{\circ}\text{C}$. ($77 \pm 3.6^{\circ}\text{F}$.) and increasing the temperature gradually at a rate not to exceed approximately 1°C . every 2 minutes. The distortion point shall be considered the temperature at which the specimen has deflected 10 mils (0.254 mm.) at the center between the supports.

The report shall include the following:

(a) The width and depth of each specimen measured at the center with a micrometer in inches or in millimeters,

(b) The distortion point in degrees Centigrade or in degrees Fahrenheit,

(c) The length of time in minutes required for each specimen to deflect 10 mils (0.254 mm.), and

(d) Any peculiar characteristics of the material as noted either during the test or after the specimen is removed from the machine.

Two curves may be plotted, one on a chart showing the minutes horizontally, the deflection in mils shown vertically to the left, and

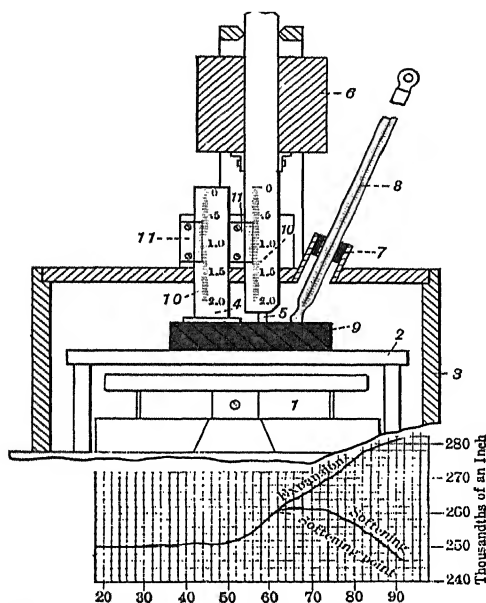


FIG. 348.—Apparatus for Determining the Softening-point of Bituminized Aggregates

the temperature in degrees shown vertically to the right. One curve represents the deflection in mils at given time intervals, and the other represents the temperature at given time intervals.

Test 56. Softening-point. An ingenious apparatus for determining the softening-point of molded insulating materials, which is likewise adapted to testing pavements, asphalt mastic floorings, expansion joints and pipe-sealing compounds, has been devised by H. R. Edgecomb³⁸ as illustrated in Fig. 348. The underlying principle consists in comparing the expansion with the tendency to soften as the temperature increases. The apparatus consists of an

electrical heater 1, a plate or slab 2 above the heater, a hood 3 for retaining the heat, a rod 4 having a relatively large lower face resting loosely on the specimen, a rod 5 having a relatively small lower face (0.01 sq. in. in area) actuated by a weight 6 of either 2.5 or 5.0 kg., and an opening 7 for the thermometer 8. The rods and thermometer rest upon the insulating material 9 to be tested, and each of the rods 4 and 5 is provided with a scale 10 operating in conjunction with stationary vernier scales 11, for recording the movement.

It is important that the sample 9 be provided with two plane faces, also that the temperature is increased at the uniform speed of 1° F. per minute. The positions of the rods 4 and 5 are noted at periodic intervals, and two curves plotted with the temperature abscissas and the movement of the rods respectively in thousandths of an inch as ordinates. These curves will be identical as the material expands throughout a certain range in temperature, but when it begins to soften, rod 5 will change its direction of travel, and instead of moving upward will embed itself in the sample. The point at which the two curves diverge represents the softening-point of the material. This is shown at 60° F. in the chart illustrated in Fig. 348.

Test 57. Resistance to Impact. Special methods have been devised for this purpose,⁴⁰ the details of which are as follows:

These methods of test are intended to determine the relative susceptibility to fracture by shock of plastic materials and electrical insulating materials as indicated by the energy expended by a standard pendulum type impact machine in breaking a standard specimen in one blow.

There are two types of pendulum impact machines and related methods of test, which use different specimens and differ in the method of holding and striking the specimen. Each specimen and method has characteristics that may dictate its use. Results by the different methods cannot be directly compared, because impact values determined by the two methods may be numerically different.

Method I is the cantilever beam or Izod type test in which the specimen is held as a cantilever beam (usually vertical) and is broken by a blow delivered at a fixed distance from the edge of the specimen clamp. The test requires a notched specimen in all cases.

The notch is intended to produce a standard degree of stress concentration.

Method II is the simple beam or Charpy type test in which the specimen is supported as a simple beam (usually horizontal) and is broken by a blow delivered midway between the supports. In this test the specimen may be either plain or notched, as required by the characteristics of the material tested.

(Method I) Cantilever Beam (Izod Type)

The machine for method *I* shall be of the pendulum type as shown in Fig. 349, of rigid construction, and accurate to 0.01 ft.-lb. for readings of less than 1 ft.-lb. and to 1 per cent for higher values. Accurate correction shall be made for friction and windage losses.

The dimensions of the machine shall be such that the center of percussion of the striker is at the point of impact, that is, the center of the striking edge.

NOTE.—The distance from the axis of support to the center of percussion, l , may be determined experimentally from the period of oscillation of the pendulum through a small angle by means of the expression $l = 0.81 P^2$, where P is the time in seconds of a complete swing (to and fro).

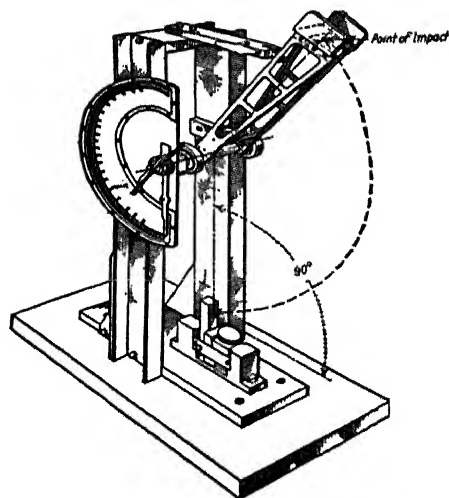
The pendulum shall be released from such a position that the linear velocity of the center of the striking edge at the instant of impact shall be approximately 11 ft. per sec., which corresponds to an initial elevation of this point of 2 ft.

The striking edge of the pendulum shall be a circular cylindrical surface of $\frac{1}{32}$ -in. radius, with its axis horizontal. The cylindrical surface shall be, when the pendulum is hanging free, tangent to the specimen in a line 0.866 in. above the top surface of the vise. The pendulum above the cylindrical portion of the striking edge shall be recessed or inclined at a suitable angle so that there is no chance of its coming in contact with the specimen during the break.

Means shall be provided for clamping the specimen rigidly in position with the edges of the supporting surfaces at 90-deg. angles.

Means shall be provided for determining the impact value of the specimen, which is the energy expended by the machine in breaking the specimen. This value is equal to the difference between the

energy in the pendulum blow and the energy remaining in the pendulum after breaking the specimen, after suitable correction has been made for windage and friction.

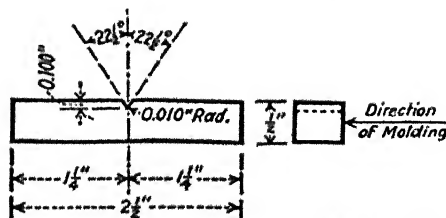


Courtesy A.S.T.M.

FIG. 349.—Cantilever Beam (Izod) Impact Machine.

The test specimen shall conform to the dimensions shown in Fig. 350. To insure duplication of the correct contour of the specified notch, all specimens shall be notched with a special formed milling cutter or other equivalent means and in such manner that the bottom of the notch is smooth, straight, and free of scratches.

For molded material the specimen shall be $\frac{1}{2}$ in. by any dimension of $\frac{1}{2}$ in., or less, agreed upon as representative of the cross-section in which the particular material is to be used. For all specimens having one dimension less than $\frac{1}{2}$ in., the notch shall be cut in the narrower side. For all compression molded specimens, the notch shall be in the side parallel to the direction of application of the molding pressure.



Courtesy A.S.T.M.

FIG. 350.—Cantilever Beam (Izod) Impact Test Specimen.

For sheet material, the thickness shall be the thickness of the sheet, except that it shall not exceed $\frac{1}{2}$ in. Sheet material thicker

than $\frac{1}{2}$ in. shall be machined down to $\frac{1}{2}$ in. Sheet material thicker than $\frac{1}{2}$ in. may be tested either edgewise or flatwise, as specified, and may be cut from the sheet either lengthwise or crosswise, as specified (Note). When specimens are tested flatwise, the notch shall be made in the original surface.

NOTE.—In referring to the cutting of the specimens of laminated sheet materials and the application of the load, the following descriptions of terms apply:

Flatwise.—Load applied to the flat side of the original sheet or plate.

Edgewise.—Load applied to the edge of the original sheet or plate.

Lengthwise.—In the direction of the length of the sheet.

Crosswise.—In the direction at right angles to the direction of the length of the sheet.

When the sheet has the same length and width, one dimension shall arbitrarily be designated as the "A" direction and the other as the "B" direction.

When the individual specimen is less than $\frac{1}{2}$ in. in thickness, the test specimen may be a composite specimen consisting of a number of individual thin pieces aggregating as nearly as possible $\frac{1}{2}$ in. in thickness. The individual members of the test specimen shall all be accurately aligned with each other and shall be tested edgewise. Single specimens less than $\frac{1}{2}$ in. in thickness may be used, provided the width is sufficient for firm, accurate clamping and the impact value of the material is sufficiently high to be accurately determined by a machine of the capacity used.

At least five individual determinations of impact value shall be made under the conditions prescribed.

The test specimen shall be rigidly clamped with the center line of the notch on the level of the top of the clamping surface (Note) and the blow shall be struck on the notched side.

NOTE.—It is recommended that a jig or template be used to locate the specimen in the jaws, as specified.

When a composite specimen is used, the individual members shall be held closely in contact and accurately aligned with each other when clamped.

The report shall include the following:

(a) A statement indicating the size of specimen, the method of test and type of preconditioning used, and, for sheet materials, the direction of testing and whether the specimens were cut lengthwise or crosswise from the sheet,

(b) The value of energy expended in breaking each individual specimen expressed in foot-pounds per inch of notch, determined by dividing the energy in foot-pounds expended in the individual test by the actual dimension in inches along the notch of the specimen broken in each test, and whether a single or a composite specimen was used, and

(c) The average of the values given in item (b), the average thickness of the individual specimen, and the number of such specimens broken in each operation of the machine.

(Method II) Simple Beam (Charpy Type)

The machine for method II shall be of the pendulum type as shown in Fig. 351, of rigid construction, and accurate to 0.01 ft.-lb.

for readings of less than 1 ft.-lb. and to 1 per cent for higher values. Accurate correction shall be made for friction and windage losses.

The dimensions of the machine shall be such that the center of percussion of the striker is at the point of impact, that is, the center of the striking edge.

The pendulum shall be released from such a position that the linear velocity of the center of the striking edge (center of percussion) at the instant of impact shall be approximately 11 ft. per sec., which corresponds to an initial elevation of this point of 2 ft.

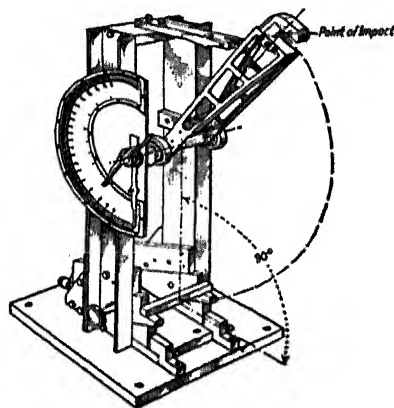


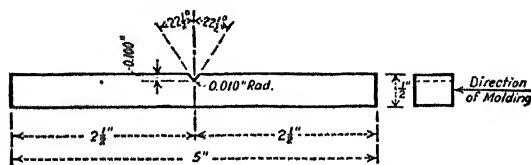
FIG. 351.—Simple Beam (Charpy) Impact Machine.

The striking edge of the pendulum shall be tapered to have an included angle of 45 deg. and shall be rounded to a radius of 0.125 in. It shall be so aligned that in the case of rectangular specimens it will make contact across the full width of the specimen.

The test specimen shall be supported against two rigid blocks in such a position that its center of gravity shall lie on a tangent to the arc of travel of the center of percussion of the pendulum drawn at the position of impact. The edges of the blocks shall be

rounded to a radius of 0.125 in. and the points of support shall be 4 in. apart.

Means shall be provided for determining the impact value of the specimen, which is the energy expended by the machine in breaking the specimen. This value is equal to the difference between the energy in the pendulum blow and the energy remaining in the pendulum after breaking the specimen, after suitable correction has been made for friction and windage.



Courtesy A.S.T.M.

FIG. 352.—Simple Beam (Charpy) Impact Test Specimen.

The test specimen shall conform to the dimensions shown in Fig. 352. The notch, when used, shall be milled with a special formed milling cutter, or machined by other equivalent means to insure duplication of correct contour and in such manner that the bottom of the notch is smooth, straight, and free of scratches.

For molded material, the specimen shall be $\frac{1}{2}$ in. by any dimension of $\frac{1}{2}$ in. or less agreed upon as representative of the cross-section in which the particular material is to be used. For all specimens having one dimension less than $\frac{1}{2}$ in., the notch, when used, shall be cut in the narrower side. For all compression molded specimens, the notch shall be in the side parallel to the direction of application of the molding pressure.

For sheet material, the thickness shall be the thickness of the sheet, except that it shall not exceed $\frac{1}{2}$ in. Sheet material thicker than $\frac{1}{2}$ in. shall be machined down to $\frac{1}{2}$ in. Sheet material thicker than $\frac{1}{2}$ in. may be tested either edgewise or flatwise, as specified, and may be cut from the sheet either lengthwise or crosswise, as specified. When specimens are tested flatwise, the notch, when used, shall be made in the original surface.

When the individual specimen is less than $\frac{1}{2}$ in. in thickness, the test specimen may be a composite specimen consisting of a number of individual thin pieces aggregating as nearly as possible $\frac{1}{2}$

in. in thickness. The individual members of the test specimen shall all be accurately aligned with each other and shall be tested edgewise. Single specimens less than $\frac{1}{2}$ in. in thickness may be used, provided the width is sufficient to insure stability during the test and the impact value of the material is sufficiently high to be accurately determined by a machine of the capacity used.

At least five individual determinations of impact value shall be made under the conditions prescribed.

The test specimen shall be supported against the steel blocks so that the blow will be struck at the center of the specimen, and on the side opposite the notch for notched specimens.

When a composite specimen is used, the individual members shall be closely in contact and accurately aligned with each other.

The report shall include the following:

(a) A statement indicating the size of specimen, the method of test and type of preconditioning used, and, for sheet materials, the direction of testing and whether the specimens were cut lengthwise or crosswise from the sheet,

(b) The value of energy expended in breaking each individual specimen expressed in foot-pounds per inch of notch, or per inch of width of the face of the specimen against which the hammer strikes, determined by dividing the energy in foot-pounds expended in the individual test by the actual dimension in inches along the notch or face of the specimen broken in each test; also, whether a single or a composite specimen was used, and

(c) The average of the values given in item (b), the average thickness or diameter of the individual specimen, and the number of such specimens broken in each operation of the machine.

Test 57a. Special Tests for Asphalt Battery Boxes.^{89a}

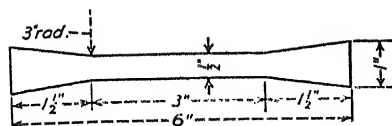
I. Tensile Strength and Elongation:

Tensile strength and elongation shall be determined on a power-driven apparatus conforming to the requirements prescribed in A.S.T.M. Designation: D 530 with the exception that the rate of travel of the power-actuated grip shall be 0.2 in. per min. instead of 0.4 in. per min.

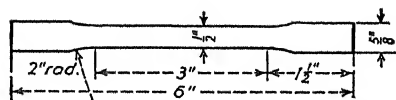
NOTE.—To secure comparable results between different testing machines, the rate of loading should be the same for each. A rate of loading of approximately 3 lb. per sec. is satisfactory.

Test specimens shall be cut in the vertical direction by a band saw or similar means from the partitions of the container and shall be taken at least 1 in. from the top of the partition. The standard specimen shall conform in shape to Fig. 352A, although a variation in width of plus 0.005 or minus 0.025 in. shall be permitted. The variation in width of an individual specimen shall not be greater than 0.005 in. The specimens shall be tested without grinding the top and bottom faces; only the contour shall be cut to suitable template dimensions.

Certain types of containers have partitions that are grooved in such a way that a standard tension test specimen cannot be cut



(a) Standard Tension Test Specimen.



(b) Alternative Tension Test Specimen

Courtesy A.S.T.M.

FIG. 352A.

from them. In such cases, the specimen used in testing hard rubber as shown in Fig. 352A or a similar specimen may be used. The size and shape of the specimen shall be included in the report.

NOTE.—The fact that results obtained from testing specimens which are not of the standard size and shape are not comparable should be noted and agreed upon by the purchaser and the manufacturer.

Two parallel gage lines 3 in. apart, for use in determining elongation, shall be scratched on the reduced section of the specimen by means of a sharp-pointed instrument such as a pair of dividers. The lines shall be perpendicular to the longitudinal axis of the specimen, one on each side of the center and 1.5 in. therefrom. The marks shall be as fine as possible, consistent with legibility, and shall not be deep enough to cause a weakening of the specimen. If it becomes necessary to use a shorter test specimen, the gage lines shall be brought nearer together and the calculation for percentage elongation changed accordingly.

The measurement, aging, and conditioning of the specimens and the temperature of test shall conform to the requirements prescribed in A.S.T.M. Designation: D 530.

Five specimens from each sample shall be tested for determination of tensile strength and elongation. Results on specimens that break outside the straight reduced section or on specimens that are obviously defective shall be discarded and retests shall be made. Such retests may, however, be omitted if the test results conform to the specification requirements. For routine testing, three specimens have usually been found adequate.

The test specimen shall be placed in the grips, using care to adjust it symmetrically in order that the tension shall be distributed uniformly over the cross-section. A tension load shall be applied to the specimen by the power-actuated grip, which shall travel at the specified rate of speed until rupture takes place. During the application of the load the distance between the gage marks on the specimen shall be noted continuously by means of dividers, so that the distance at the instant of rupture may be determined within 0.01 in. (Note). This value shall be recorded for use in calculating the ultimate elongation. After rupture of the specimen, the breaking load in pounds shall be read and recorded together with the original minimum width and thickness of the specimen so that the tensile strength may be calculated.

NOTE.—The measurement of elongation by means of an extensometer is not to be recommended unless the extensometer action is independent of the movement of the testing machine and unless the breaking strength is unaffected by reason of the attachment of the extensometer to the test specimen. For control purposes, such instruments may be found to be very acceptable.

The tensile strength and elongation shall be calculated in accordance with A.S.T.M. Designation: D 530.

The report shall include the following:

- (a) Date of test,
- (b) Temperature of test room,
- (c) Type of testing machine,
- (d) Description of sample and type of test specimen,
- (e) The observed and recorded data, and
- (f) The results calculated.

II. Bulge Test:

The purpose of this test is to measure the tendency of a battery container to soften and bulge due to the heat generated when it is in service.

The apparatus shall consist of the following:

(a) Electrical Equipment: Means for passing an electric current through the cells of the container, when filled with an electrolyte.

(b) Temperature Control: A control for maintaining constant temperature of the electrolyte during test.

(c) Measuring Device: A device for measuring the length and width of the container.

The width and length of the battery container from the middle point of opposite panels shall be measured to the nearest $\frac{1}{64}$ in. Each cell of the container shall be filled to a point approximately $\frac{3}{8}$ in. from the top with water to which sufficient sulfuric acid has been added to give a specific gravity of approximately 1.01. The electrolyte shall be heated to a temperature of $160 \pm 2^\circ \text{F.}$ before it is placed in the container.

Carbon or lead electrodes shall be placed in the cells with the necessary connections to provide for the passing of a continuous current. After the initial cooling when the electrolyte is placed in the container, the temperature shall be raised to $160 \pm 2^\circ \text{F.}$ by means of an electric current and that temperature maintained by suitable electrical control for a period of 3 hr.

After the container and acid have cooled to the temperature of the room, which should be maintained at $75 \pm 5^\circ \text{F.}$, the width and length shall be measured again and the changes recorded.

The report shall include the following:

(a) The original length, the length after test, and the increase in length, and

(b) The original width, the width after test, and the increase in width.

NOTE.—When two or more containers are tested at a time, a space of at least 2 in. shall be left between them to permit proper circulation of air.

III. Sensitivity to Hot-cold Cycles:

This test is designed to measure the tendency of a battery container to develop cracks as a result of abrupt and severe changes in temperature.

The apparatus shall consist of the following:

(a) Cold Chamber: A refrigerator or cold room of sufficient capacity to produce the temperatures required.

(b) Hot Chamber: A hot chamber or room with heating means sufficient to produce the temperatures required.

Each cell of the battery container shall be filled to a point 0.5 to 1.0 in. from the top with battery acid (sp. gr. 1.30 at 77° F.). Place the container in the refrigerator, which shall be maintained at a temperature of $0 \pm 2^\circ$ F. for a period of 16 hr. The capacity of the refrigerator shall be sufficient to reduce the temperature of the acid in the middle cell of the container to $0 \pm 2^\circ$ F. in 13 to 15 hr.

Upon withdrawal of the container from the refrigerator, it shall be placed directly into the hot chamber. The hot chamber shall have a temperature between 110° and 120° F. and shall be of such capacity as to cause the acid in the middle cell of the container to reach $83 \pm 2^\circ$ F. in 5 hr.

After 5 hr. in the hot chamber, the container shall be withdrawn and allowed to rest 3 hr. at room temperature, which will complete the first cycle of 24 hr. Not only initial and final temperatures, but also the intermediate temperatures, shall be approximated.

Examination of the container shall be made to note the development of any cracks and leaks, and the cycle shall be repeated until such time as cracks may develop, or until the required number of cycles have been completed.

NOTE.—Precautions must be taken, when several containers are to be tested at one time, to enable each container to reach the required temperature levels by proper spacing of the containers and regulation of the refrigerator and hot chamber. Circulation of the air in both the refrigerator and the hot chamber are necessary to produce uniform conditions.

The report shall include the following:

- (a) Record of the temperatures attained in each cycle, and
- (b) Number of cycles before failure occurs.

IV. Acid Absorption:

This test is intended to measure the penetration of battery acid into the partitions of a battery container at an elevated temperature.

The test specimen shall be 2 in. square and of the thickness of the partition from which it is cut. The specimen shall be taken at least 1 in. from the top of the partition, and shall not contain any irregular flow marks.

Since the rate of penetration of acid through the cut edges is different from the rate through the sides, the edges may be sealed to overcome this difficulty. A high melting mastic or other acid-proof adhesive may be used for this purpose.

Two test specimens shall be tested and the average values used in determining the acid absorption and penetration.

Weigh the test specimen and submerge it in H_2SO_4 (sp. gr. 1.30 at 77° F.) in a jar or glass container. When several test specimens are to be placed in the same container, glass triangles or other suitable means shall be used to separate the specimens from each other. Fresh acid shall be used for each new test.

Cover the jar to avoid evaporation and concentration of the acid; then place it in an oven maintained at $150 \pm 2^\circ \text{F.}$, for a period of 28 days. At the conclusion of this period, withdraw the specimen and wash it in running tap water for approximately 1 min. to remove the acid from the surface of the specimen. Wipe the surface with a cotton cloth to take up residual water, and weigh the specimen immediately.

After the test specimen is weighed, cut it through the center into two pieces, each about 2 in. in length and 1 in. in width. Polish the freshly cut edge with benzene or other suitable solvent, and measure the average penetration of the acid to the nearest $\frac{1}{64}$ in. In determining the average penetration, only the area within 0.5 in. of the middle point shall be noted.

Alternative Method: If the 28-day period for the test is impracticable, a shorter period of 7 days at $150 \pm 2^\circ \text{F.}$ may be substituted. The procedure shall otherwise be carried out as described (Note).

NOTE.—The 7-day period of immersion in acid usually results in less penetration of the acid than in the preferred test period of 28 days.

The report shall include the following:

(a) Increase in weight of the test specimen, expressed in grams (indicate whether or not the edges are sealed),

- (b) Penetration of the acid into the interior of the test specimen, expressed in sixty-fourths of an inch,
- (c) Time in acid, and
- (d) Temperature of acid.

Test 58. Electrical Tests. The A.S.T.M. has similarly standardized a series of tests for these determinations, and for the details the reader is referred to the citations given in the following references:

- (a) *Dielectric Strength:*
 - Of sheet, tape and molded insulating materials.⁴⁰
 - Of solid filling and treating compounds for insulation.⁴¹
 - Of electrical insulating oils.⁴²
- (b) *Power Factor and Dielectric Constant:*
 - Of solid insulating materials.⁴³
- (c) *Surface and Volume Resistivity:*
 - Of solid and liquid insulating materials.⁴⁴

The dielectric strength test has also been recommended for roughly ascertaining the proportion of coal-tar or coal-tar pitch in admixture with asphalt.⁴⁵

(B) SEPARATION OF FINISHED PRODUCT INTO ITS COMPONENT PARTS

SEPARATION OF THE BITUMINOUS MATTER AND DISCRETE AGGREGATE

Bituminized aggregates are separated into their bituminous and discrete components for the combined purposes of ascertaining the percentage and nature of the mineral constituents and for examining the physical and chemical characteristics of the bituminous binder, with the object of its identification or duplication. Two classes of methods are available, depending upon whether the aggregate is associated with an asphaltic or a coal-tar pitch binder.

Various methods have also been described⁴⁶ for examining small quantities of such materials, by microanalytical and other procedures. The following, however, constitute the methods generally employed:

Test 59. Methods Suitable for Aggregates Associated with an Asphaltic Binder. Two methods have been devised, one based

upon the use of hot extractor, and the other upon the use of a centrifugal extractor.

Test 59a. Hot Extraction Method. I. Alternate: This has been standardized as follows:⁴⁷

These methods cover the procedures for testing bituminous mastics, grouts, and like mixtures, which may be classified and defined as follows:

Bituminous Grout: A mixture of bituminous material as a binder and sandy mineral matter as an aggregate, which when heated to a suitable temperature becomes sufficiently fluid to flow into place without mechanical manipulation, and which on cooling congeals to a compact mass.

Asphalt Mastic: A mixture containing (1) asphaltic material as a binder and graded mineral matter as an aggregate, or (2) pulverized native rock asphalt (to which asphaltic material may have been added); either of which when heated to a suitable temperature may be poured into place but which requires trowelling to form it into a compact mass. (See also p. 636.)

Asphalt Mastic Cake: A mixture containing asphaltic material as a binder and an aggregate consisting chiefly of calcareous or siliceous dust cast into the form of blocks or "cakes" and adapted for use in preparing asphalt mastic.

Bituminous grouts shall be heated in an oven or on a hot plate in a pan or other suitable container at the lowest possible temperature to prevent overheating and volatilization, and when sufficiently fluid, shall be thoroughly stirred to insure a uniform sample, whereupon 10 to 30 g. shall be taken for analysis.

Asphalt mastics or mastic cake shall be warmed on a hot plate or in a hot oven until soft enough to be broken up or stirred, so that a representative sample for analysis may be taken. The amount taken for analysis will depend upon the amount of coarse gravel or stone in the mixture. The larger the gravel or stone, the larger will be the sample required for accuracy. The size of samples to be taken shall be as follows: Where all particles pass a No. 10 sieve, 10 to 30 g.; where 25 per cent of the aggregate is retained on a No. 10 sieve, 50 g.; where 50 per cent of the aggregate is retained on a No. 10 sieve, 100 g.; and where 75 per cent of the aggregate is retained on a No. 10 sieve, 200 g.

NOTE.—Where the properties of the extracted bituminous matter are to be determined, 500 g. shall be taken and extracted with pure benzol as described.

(a) *For Analysis of 10- to 30-g. Samples:** In cases where a 10- to 30-g. sample is sufficient, the analysis shall be carried out by means of the glass extractor illustrated in Fig. 325.

An ordinary fat-free Whatman or S. and S. filter thimble, 60 mm. in length by 26 mm. outside diameter, shall be dried for 30 minutes in an oven at 212° F., allowed to cool in a desiccator, and then weighed in a suitable weighing bottle. The weighed sample shall be placed in the thimble and a disc of wool felt or a plug of absorbent cotton shall be placed over the sample in the top of the thimble to distribute the solvent uniformly and prevent splashing. After placing in the extractor, 40 to 50 ml. of carbon disulfide† shall be poured over the sample, whereupon the thimble containing the sample shall be suspended under the condenser by a fine wire bail. The flask shall be cautiously heated by a steam-bath or electric heater just enough to vaporize the solvent. Cold water is circulated through the condenser. The heat evaporates the carbon disulfide in the flask. This condenses upon the condenser and drops back upon the sample through which it filters, thus dissolving out the bitumen which collects in the bottom of the flask.

The extraction should be discontinued when the carbon disulfide drops colorless from the filter. The time of extraction will depend upon the nature of the bitumen and mineral aggregate in the sample and upon the degree of heat applied, the coldness of the water in the condenser and other factors. In some cases extraction may be complete in one hour, in others four or five hours may be necessary.

When the solvent comes through clear, the filter shall be removed and washed with a fine jet of carbon disulfide from a washing bottle to wash out any bitumen that may be retained at the top of the paper and to break up any channels that may have been formed

* This method for analysis of larger samples is especially adapted for asphalt mastics, grouts, and mastic cake. If the binder is coal-tar pitch, the "free carbon" constituent of the binder will remain with the mineral aggregate.

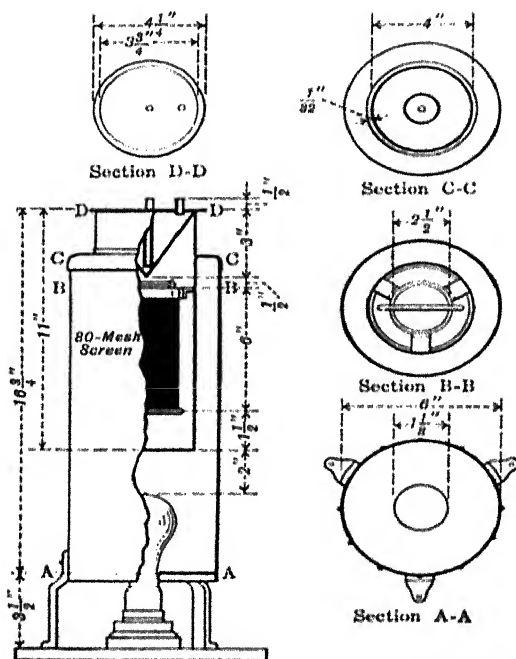
† Carbon tetrachloride, benzol, or chloroform may be used instead of carbon disulfide, with the only difference that in the case of noninflammable solvents, the solvent will have to be evaporated from the solution of bitumen to determine the ash for correction instead of burning off directly. When the solvent is expelled, the bitumen can be ignited for ash.

by the carbon disulfide passing through. If the washings show any color, the thimble shall be put back and extraction continued until the solvent again becomes colorless. It shall then be removed, dried carefully, at a low temperature at first to prevent ignition of the absorbed carbon disulfide, and finally to constant weight at 100° C. (212° F.), cooled and weighed.

The solution in the flask shall be rinsed into a weighed porcelain or silica evaporating dish or crucible and the solvent burned off under a hood. The residue shall be ignited over a flame or in a muffle and the ash weighed, and the weight added to that of the mineral matter in the filter paper. This is to correct for the fine mineral matter which will be carried through the paper by the solvent. Should there be a considerable amount of ash recovered in this way, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily, however, the mineral matter going through the paper will be so small in amount that the difference caused by ignition may be neglected. The corrected loss in weight on the original sample represents the percentage of asphalt present.

The sieve analysis of the mineral aggregate shall be made in accordance with A.S.T.M.: C 136. The extracted residue shall be transferred from the thimble to the No. 200 (74-micron) sieve, the paper being gently rubbed to free adhering particles. The aggregate on the sieve shall be gently rubbed with the fingers to break up lumps and to free any particles of fine dust that might adhere to larger sand particles. The sieve shall be shaken over a piece of paper from side to side with the right hand, striking it sharply against the palm of the left hand until no appreciable amount of dust comes through the sieve on to the paper. The paper shall be cleared from time to time by raising one side with the left hand and rolling off the siftings, so that it can be seen when the sifting is complete. The material remaining on the No. 200 sieve shall be weighed and the amount of material which has passed through the No. 200 sieve shall be determined by difference. This operation shall be repeated upon the coarser sieves in order and the amount passing each sieve and retained on the next finer recorded as percentage of the original sample.

(b) *For Analysis of 50- to 500-g. Samples:* The apparatus for analysis of samples containing coarse aggregate shall be the large extractor shown in Fig. 353, consisting of a large brass cylinder, through the bottom of which projects a 16-candlepower incandes-



Courtesy A.S.T.M.

FIG. 353.—Extraction Apparatus.

cent carbon filament bulb to supply heat to the extraction apparatus proper, which is held in the upper portion of the cylinder. This apparatus is composed of a cylindrical brass vessel for holding the solvent, a cylindrical wire basket made of 80-mesh wire cloth suspended in the cylinder, and an inverted conical condenser which serves as a top.

A large filter paper, 12 or 13 in. in diameter, shall be fitted inside the wire basket of the extractor by folding once more than in ordinary filtering, or by wrapping it over a form which fits inside

the basket (a cylindrical bottle of proper size makes a good form) and placing it inside the basket.

The basket with contained filter paper shall be dried and weighed. The sample shall be weighed and packed in the filter paper in the basket. Care should be taken not to pack all coarse particles in one place and the fine particles in another, but to have them mixed together in uniform proportions.

The sample shall be covered with a disk of felt or wad of absorbent cotton to insure even distribution of the dropping solvent, thus preventing it from forming a channel through the sample. The basket shall be suspended in the extractor and 150 to 200 ml. of carbon disulfide poured over the felt or cotton. The condenser shall be placed over the top and water circulated through it. Current shall be started through the electric lamp underneath the extractor and the extraction carried on exactly as in the smaller glass extractor, but on a larger scale. The time for extraction will vary from 3 to 12 or more hours, depending upon the nature of the sample.

To determine when extraction is complete, the condenser shall be raised and the basket lifted out to observe if drippings are clear. One or two drops caught upon white filter paper should leave but a light stain.

The drying and weighing of the basket, burning off of the solution for correction, and calculation of the weight of mineral matter shall be determined as in the foregoing, except that in sifting mineral aggregates containing coarse stone, it is well to remove the stone by putting the mineral aggregate first through a No. 10 (2000-micron) sieve, as the large stone would injure the No. 200 (74-micron) sieve.

The stone removed by the No. 10 sieve may be sieved through $\frac{1}{4}$ -in., $\frac{1}{2}$ -in., and coarser sieves in order, as described in A.S.T.M.: C 136, and the fine material passing the No. 10 sieve through No. 200 and coarser sieves in order, all amounts passing any one sieve and retained on the next finer one being reported as percentage of the original sample. Thus the ingredients of the mixture may be roughly separated by the use of the proper size sieves, and examined for physical and chemical characteristics.

NOTE.—The siftings passing the No. 200 (74-micron) sieve will consist largely of the dust or pulverized rock used in the mixture. The material between the No. 100 (149-micron) and the No. 10 (2000-micron) will consist largely of the sand used in the mixture, with any particles of fine crushed stone within these limits that existed in the original materials. Gravel or broken stone as a rule will be larger than No. 10 or No. 8 (2380-micron).

When the mastic is to be used for acid-proof floors, tank linings, etc., pulverized silica and other materials insoluble in acid are used in preparing the mixture. The most important chemical property, therefore, is solubility or insolubility in mineral acids. This determination may be carried out in the following manner:

Dry to constant weight at 325° F. about 2.5 g. of the dust, or of the coarser material, or stone which has been previously pulverized in a mortar. The loss will represent any moisture that may have been retained in the material as well as any absorbed solvent that may not have been expelled while drying the sample after extraction. Place 1 g. of the dried sample in a 250-ml. beaker, cover with a watch glass and add 10 ml. of C.P. HCl (sp. gr. 1.19), dilute with 50 ml. of distilled water. Break up any lumps by means of a glass stirring rod. Note any effervescence which may occur. If considerable effervescing takes place, the sample may be said to be carbonate. If all the sample, or practically all, dissolves, the determination need not be carried further. If apparently insoluble, heat it carefully and boil for 15 min., and filter through a tared Gooch filter, and wash the insoluble residue with hot water. Ignite and weigh and report the loss as the percentage soluble in hydrochloric acid.

Note that any water in chemical combination with the mineral constituents is not ascertained by this procedure, but the amount present is usually so small as to be negligible.

II. Alternate: The following rapid method has been proposed:⁴⁸

Weigh 15 g. of sand-asphalt mixture in a 12.5-cm. filter-paper in a small brass mesh basket (50- to 100-mesh) suspended by hooks from a cork inside a 750-ml. Erlenmeyer flask containing 100-ml. xylene or toluene, as illustrated in Fig. 354. The edges of the filter-paper are turned inward to prevent the creeping over or splashing of fine mineral matter. Water is collected in a Dean-Stark tube below the reflux condenser. Greater accuracy is ob-

tained in the determination of the water content if a separate 100-g. sample is extracted in a Dean-Stark apparatus, in which case approximately $1\frac{1}{2}$ hours are required to complete the extraction. Pyridine is recommended for extracting sand-tar mixes, especially where changes in the tar have resulted through: (a) oxidation; (b) polymerization; or (c) reaction with any lime that may be incorporated in the mix. 5–8 g. of sand-tar mix are boiled with 30 ml. pyridine and decanted through a weighed Gooch crucible and washed with boiling pyridine until the filtrate is clear. The insoluble matter left on the mineral aggregate after extraction with CS_2 , CHCl_3 , C_2HCl_3 , C_6H_6 , etc., has been designated "organic insolubles," the quantity of which depends upon the particular solvent used and upon the specific absorptive properties of the aggregate. The following figures were obtained upon extracting a native asphalt with the following solvents, using the hot extraction method: CCl_4 1.51%; C_6H_6 1.23%; CS_2 1.20%; C_2HCl_3 1.20%; CHCl_3 0.73%; $\text{C}_5\text{H}_5\text{N}$ 0.55%.⁴⁰

Test 59b. Cold Extraction Method.

I. Alternate: Weigh out 1000 g. of the well-disintegrated sample into an 800 ml. beaker and add 500 ml. carbon disulfide. Stir five or six times during the first 24 hours thoroughly to break up the aggregate, and then let stand quietly for 48 hours, after which the solution shall be carefully decanted and filtered through a series of three 100 ml. funnels, using absorbent cotton as the filtering medium. The aggregate left in the beaker shall be subjected to a second washing, conducted as described. The extract shall be combined and placed in a quart jar, which shall be tightly covered and allowed to stand quietly for 48 to

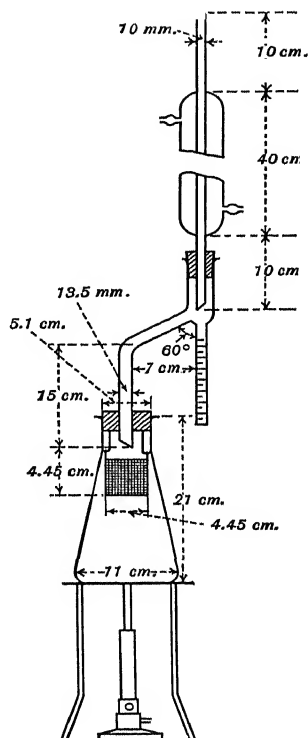


FIG. 354.—Rapid Hot Extraction Apparatus.

72 hours, after which the solution shall be filtered through an asbestos mat in a Gooch crucible to remove the last traces of sediment. The mineral ingredients shall then be dried and weighed as previously described.

II. Alternate: In the case of mastic block and similar bituminized aggregates containing a high percentage of finely-divided mineral matter, there is apt to be a loss of fine material, caused by the carbon disulfide solution creeping behind the folds of the

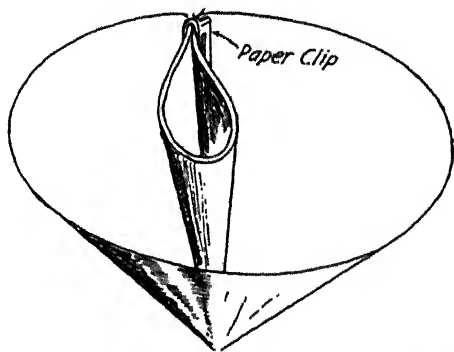


FIG. 355.—Method of Folding Filter-papers for Extraction Tests.

filter paper and over its edge, rather than through the paper. In the case of mastic the following procedure is recommended: ⁶⁰ Two 32-cm. No. 5 Whatman filter papers, after being dried in an oven and cooled in a desiccator, are counterpoised. These papers are folded together and fastened with a paper-clip in the manner shown in Fig. 355; whereupon they are placed in a glass funnel 7 in. in diameter, the top edge of which is ground flat. The funnel is covered with an 8-in. diameter glass plate in the center of which a $\frac{5}{8}$ -in. diameter hole has been drilled. To make certain that the plate completely closes the funnel, it is advisable to secure the plate to the funnel with gelatine cement, prepared by dissolving 10 g. of gelatine in 80 ml. water and adding 20 g. glycerin. The mastic block is warmed in an oven and broken into small pieces. Two 75 g. representative samples should be taken and run in duplicate. One sample is introduced into the counterpoised filter and the funnel

covered with the glass plate as described. Carbon disulfide is introduced directly into the funnel through a 1,500 ml. separatory funnel as shown in Fig. 356. The addition of carbon disulfide may be made automatic, so that when the level of the liquid in the filter falls below a predetermined height, a fresh supply will be admitted, and this may continue overnight without attention. When the filtrate comes through colorless, the carbon disulfide supply is stopped and the paper allowed to dry, first in air, and finally in an oven at 120°C . for one hour. After cooling in a desiccator, the filter and contents are weighed, the outside paper being placed on the weight-pan as a counterpoise. The weight of asphalt extracted is ascertained by subtracting the weight of the aggregate from the weight of the sample taken, and its percentage calculated.

For products other than mastics, the following weights of material are recommended: surfacings containing aggregate all passing 10-mesh, two 50 g. samples; surfacings containing up to 25 per cent of $\frac{1}{4}$ -in. or $\frac{3}{8}$ -in. aggregate, two 150 g. samples; surfacings containing up to 25 per cent $\frac{3}{4}$ -in. aggregate, two 300 g. samples; surfacings containing up to 50 per cent $\frac{3}{4}$ -in. aggregate, two 500 g. samples.

III. Alternate: Consists⁶¹ in covering the coarsely broken sample with carbon disulfide and allowing same to stand quietly overnight, whereupon it will be found that most of the soluble material has gone into solution. The residue is then washed with 8 to 10 portions of carbon disulfide. Fine mineral matter is removed from the combined extracts by filtering or centrifuging.

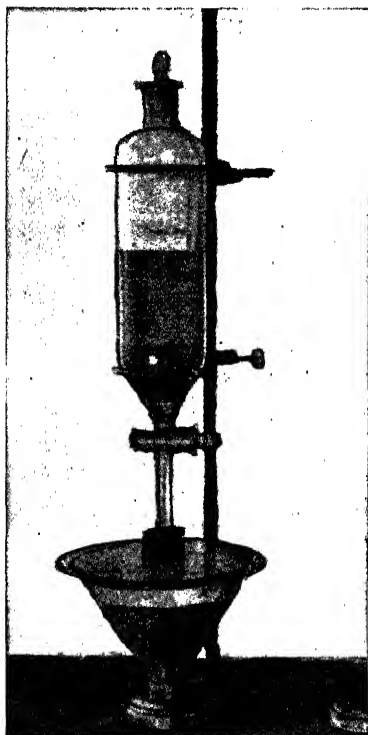


FIG. 356.—Apparatus for Cold Extraction Test.

Test 59c. Centrifugal Extraction Method. I. Alternate: The most efficient apparatus of this type was designed by C. S. Reeve,⁶² as illustrated in Figs. 357 and 358. It consists of a $\frac{1}{8}$ h.p. vertical motor *a*, capable of making 1100 revolutions per minute at 110 volts, with either direct or alternating current. Its shaft projects into a cylindrical copper vessel *b*, having a concave bottom and draining into the spout *c*. A circular brass plate *d*, $9\frac{1}{2}$ in. in diameter, supports an inverted iron bowl *e*, $8\frac{1}{2}$ in. in diameter by

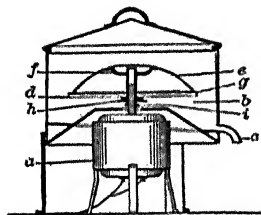
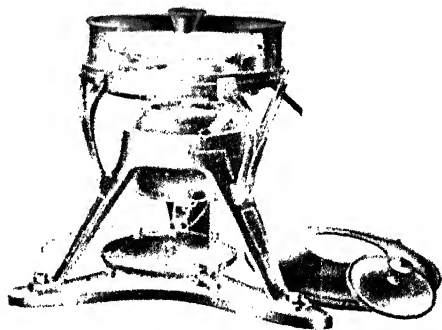


FIG. 357.—Centrifugal Extractor.



Courtesy of Braun Corp.

FIG. 358.—Centrifugal Extractor.

$2\frac{5}{16}$ in. high, having a 2-in. circular hole at the top. A brass cup *f* is fastened to the inner side of the bowl, having a circle of $\frac{1}{8}$ -in. holes for the admission of solvent, and terminating in a hollow axle which fits snugly through a hole in the center of the brass plate *d*. A felt ring *g*, $\frac{3}{4}$ in. wide and about 0.090 in. thick (cut from No. 80 roofing felt) is firmly pressed against the bowl by the milled nut *h* for which the hollow axle is suitably threaded. The axle in turn fits snugly over the shaft of the motor, to which it is secured by a slot and cross-pin.

Procedure: The filter ring shall be dried for 30 minutes in an oven at 212° F., allowed to cool in a desiccator and then weighed. The required quantity (10 or 25 g.) of the disintegrated sample shall be accurately weighed on a tared watch glass or weighing scoop. The sample shall then be placed in the bowl of the rotarex machine, care being taken to evenly distribute the mixture around

the periphery of the bowl to prevent vibration during the extraction. The filter ring and bowl plate shall then be placed on the bowl and fastened down fairly tight. The bowl shall then be placed in position in the rotarex machine and from 40 to 50 ml. of carbon disulfide poured into the bowl through the funnel opening. After allowing the material to digest for about 15 minutes, the motor shall be started, slowly at first, and then the speed increased sufficiently to cause the dissolved bitumen to flow from the spout in a thin stream. The dissolved bitumen or filtrate shall be collected in a beaker or other receptacle. When the first charge has drained, the motor shall be stopped and a fresh charge of carbon disulfide added and again allowed to digest for 15 minutes and the machine started as before. This operation shall be repeated from three to five times or until the solvent runs clear from the spout, showing all bitumen has been dissolved from the sample, this usually requiring three to four washings.

When the last addition of solvent has drained off, the bowl shall be removed and placed with the bowl plate and filter ring on a sheet of glazed manila paper and allowed to dry at room temperature. The aggregate shall then be brushed from the bowl onto the paper together with any aggregate adhering to the bowl plate and filter ring. The aggregate shall then be transferred to a tared watch glass or pan and together with the filter ring dried in an oven at 212° F., cooled in a desiccator and weighed.

In making the correction for the mineral matter suspended in the filtrate, add sufficient carbon disulfide to bring the same to a definite volume and a multiple of 100 ml. The whole filtrate shall then be thoroughly shaken so that the mineral matter is uniformly dispersed throughout the filtrate and an aliquot part of 100 ml. then taken and transferred into a weighed porcelain or silica evaporating dish and the solvent burned off under a hood. The residue shall then be ignited over a gas flame or in a muffle until it is entirely free from carbon, cooled and weighed and the total ash correction determined as follows:

$$\text{Total ash} = \frac{\text{Total filtrate in milliliters} \times \text{Ash in grams recovered in aliquot part}}{\text{Aliquot part in milliliters}}$$

Should there be a considerable amount of ash recovered in this manner, and if it is found that the mineral matter is calcium or other carbonate, it should be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat.

II. Alternate: This consists in using a centrifugal tube in the following manner: The apparatus to be used in this test shall be any suitable type of centrifugal machine capable of being rotated rapidly (1800 r.p.m.), surmounted by a head carrying an equal number of metal tube shields, slightly larger than the glass tubes used, and provided with rubber cushions, the whole being encased in a metal shield. In place of glass tubes aluminum or bronze tubes may be used.

Procedure: The required quantity (10 or 25 g.) of the disintegrated sample selected at random throughout the sample shall be accurately weighed on a tared watch glass or weighing scoop. After weighing, the sample shall be transferred to a numbered and weighed tube. The tube shall be filled with a definite quantity of carbon disulfide approximately three-fourths full and then inserted in the metal tube shield of the centrifuge machine. The tubes shall be so placed in the centrifuge machine as to have equal weights opposite each other around the head of the centrifuge. After allowing the material in the tube to digest for about 15 minutes, the centrifuge machine shall be started, slowly at first, gradually bringing it up to full speed and whirling from 5 to 10 minutes. The machine shall then be stopped, the tube removed and the solvent and dissolved bitumen decanted into a numbered flask. The tube shall again be filled with carbon disulfide, mixing the solid matter at the bottom of the tube with the solvent by stirring, the material allowed to digest for 15 minutes, whirled from 5 to 10 minutes, the solvent decanted and the bitumen dissolved as before. This operation shall be repeated from three to five times, or until the solvent in the tube, after whirling from 5 to 10 minutes, is clear and colorless.

The tube shall now be removed from the machine and the solvent allowed to evaporate from the aggregate at room temperature and the material then dried in an oven at 212° F. to constant weight, cooled in a desiccator and weighed.

The solvent and dissolved bitumen in the flask shall be rinsed

into a weighed porcelain or silica evaporating dish and the solvent burned off under a hood. The residue shall then be ignited over a gas flame or in a muffle until it is entirely free from carbon, allowed to cool in a desiccator and the ash weighed and its weight added to the weight of the mineral matter in the tube. Should there be a considerable amount of ash recovered in this manner, and if it is found that the mineral matter is calcium or other carbonate, it shall be recarbonated by repeated treatment with ammonium carbonate solution and finally ignited at a dull red heat. Ordinarily the mineral matter in the solution is found to be so small that the difference caused by ignition may be neglected.

Test 60. Method Suitable for Aggregates Associated with Coal-tar Pitch Binder. *I. Alternate:* An apparatus has been devised as illustrated in Fig. 359,⁵⁸ consisting of a 2-liter copper still fitted to a metal cover of funnel shape, and fastened thereto with six bolts. A flanged glass tube about 1.5 in. in diameter is fastened to the cover by means of a union pipe-joint, as shown. In running the test, 2000 g. of the bituminized aggregate are placed in the still, the cover fastened in place and sufficient sodium carbonate solution (sp. gr. at 60° F. of 1.27 to 1.28) added to bring the surface of the liquid just visible within the glass tube. Gradually apply heat to the bottom of the still. As the coal-tar pitch liquefies, it rises in globules until it accumulates in a layer in the upper portion of the glass tube. Care should be taken not to heat the liquid higher than necessary to float the pitch. Wash down the reflex condenser with 10 ml. of soda solution to recover adhering oils. Then stopper the upper end of the condenser and remove the clamp from the overflow tube. Heat the bottom of the still rapidly to form a steam pocket below the false bottom, whereupon the liquid pitch will be ejected through the overflow tube and caught in a separate container. Centrifuge the pitch for fifteen minutes to remove all the sodium carbonate solution, and retain it for further examination. If any difficulty is experienced in floating the pitch, as may be the case with old or weathered samples, add 100 g. of solid sodium carbonate at the start of the test. This method will recover all the free carbon with the pitch and separate the mineral aggregate in a clean state.

II. Alternate: A centrifugal method has been proposed for separating binders from road compositions in admixture with aggregates, so that the separated binder may readily be examined by any appropriate test described in Chapter XXXII.⁵⁴ It is claimed that the binder may be recovered without alteration, and that the

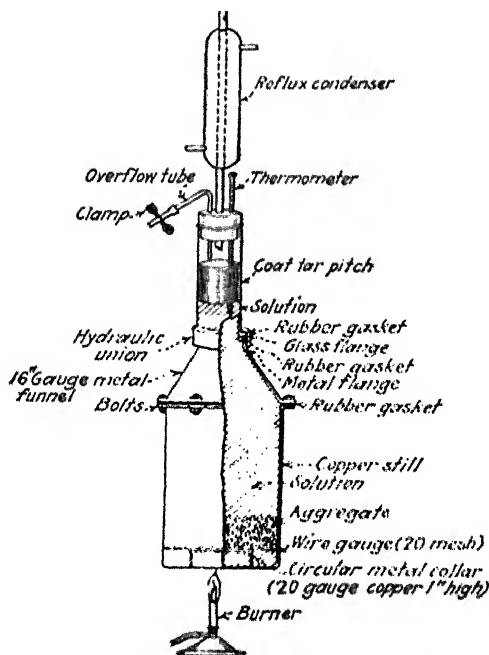


FIG. 359.—Extractor for Coal-tar Pitch Aggregates.

method is especially suitable in the case of binders: (a) containing a substantial proportion of volatile constituents, or (b) tar binders which are not completely soluble in any known solvent.

The centrifuge is illustrated in Fig. 360, and carries four receptacles held in place by three-pronged spring clips, which rotate in a vertical plane at speeds up to 1,200 r.p.m. and at temperatures up to 120° F. Each receptacle consists of a cylindrical cup (A) provided with a cover (B) and a lower thimble (C) which may be unscrewed. The lower conical end of the cup carries a

metal grid supporting a 200-mesh gauze filter (D) which is held in place by a spring clip. The centrifuge is enclosed in a casing which is heated by steam or electrical units installed at the base.

A representative sample of the road mixture is warmed and 100 g. introduced into the cup (A) over the gauze (D). The interior is heated to 100–120° F. and the receptacles are first rotated at low speed for 1 hour, whereupon the speed is increased

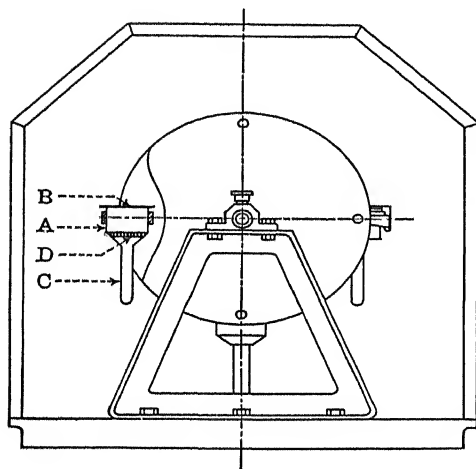


FIG. 360.—Centrifugal Separator.

to 1,200 r.p.m. for $\frac{1}{2}$ hour. The thimbles (C) are unscrewed and the separated binder poured off for further examination. During the spinning, the binder is separated from the aggregate and passes through the filter into the thimble where any fine filler is caused to collect at the bottom through centrifugal action. It should be noted, however, that any free carbon present in road tars will similarly be removed.

Another method⁵⁵ consists in first extracting the bituminized aggregate with carbon disulfide, drying and weighing the residue, which is then boiled for $\frac{1}{2}$ hour with 3 to 5 per cent potassium soap solution, with constant stirring. Decant through a fine mesh screen, wash the residue with water until free from soap, dry the mineral matter and calculate the free carbon by difference.

Another procedure⁵⁶ consists in extracting the sample with car-

bon disulfide, drying and weighing the residue. The latter is then tested in two ways, as follows: (1) A portion is heated with 10 per cent HCl, and the CO_2 liberated from any carbonates present in the mineral aggregate is caught and weighed. (2) Another portion is heated with a saturated solution of CrO_3 in concentrated H_2SO_4 and the CO_2 liberated from the carbonates plus that derived from the non-mineral constituents recovered and weighed. The difference between (2) and (1) represents the CO_2 liberated from the non-mineral constituents, which have been found to carry an average of 85 per cent of carbon by weight. The percentage of non-mineral constituents present in the extracted mineral aggregate may be calculated from the following formula:

$$\frac{\text{Per cent (2)} - \text{per cent (1)} \times 0.2727 \times 100}{85}$$

(C) RECOVERY AND EXAMINATION OF EXTRACTED BITUMINOUS MATTER

Test 61. Separation of Bituminous Constituents. The bituminous constituents may be separated from the solvent used for extraction by any of the procedures described in Test 21*b*. They may thereupon be examined by means of the tests given in Chapter XXXII.

Methods have also been described⁵⁷ for examining mixtures of bituminous substances with rubber (e.g., electrical insulating materials, molded articles, rubber substitutes, etc.).

(D) EXAMINATION OF THE SEPARATED AGGREGATE

Inorganic Aggregates:

These will include the coarse mineral matter, as well as the fine mineral matter (colloidal), retained on the Gooch filter. After drying, it should be rubbed gently with the fingers to break up lumps and to free any particles of fine dust that might adhere to the larger particles.

NOTE.—The presence of any non-mineral matter insoluble in carbon disulfide will be revealed by the discoloration of the mineral particles. In this case, the weight of the latter should be corrected by igniting it until all carbonaceous matter is destroyed, and then reweighing.

TABLE CXLIX

NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES

Size of Sieve Designation	Sieve Opening		Permissible Variations in Average Opening, per cent	Permissible Variations in Maximum Opening, ^a per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
(1)	(2)	(3)	(4)	(5)	(6)	(7)
COARSE SERIES						
(4.75-in.) ^b	107.6	4.24	±2	±3	5.6 to 9.7	0.220 to 0.380
4-in.	101.6	4.00	±2	±3	5.6 to 9.7	0.220 to 0.380
3½-in.	88.9	3.50	±2	±3	5.3 to 9.3	0.210 to 0.365
3-in.	76.2	3.00	±2	±3	4.8 to 8.1	0.190 to 0.320
2½-in.	63.5	2.50	±2	±3	4.4 to 7.1	0.175 to 0.280
(2.12-in.) ^b	53.8	2.12	±2	±3	4.1 to 6.2	0.160 to 0.245
2-in.	50.8	2.00	±2	±3	4.1 to 6.2	0.160 to 0.245
1¾-in.	44.4	1.75	±2	±3	3.8 to 5.7	0.150 to 0.225
1½-in.	38.1	1.50	±2	±3	3.7 to 5.3	0.145 to 0.210
1¼-in.	31.7	1.25	±2	±3	3.5 to 4.8	0.140 to 0.190
(1.06-in.) ^b	26.9	1.06	±3	±5	3.43 to 4.50	0.135 to 0.177
1-in.	25.4	1.00	±3	±5	3.43 to 4.50	0.135 to 0.177
¾-in.	22.2	0.875	±3	±5	3.23 to 4.22	0.127 to 0.166
¾-in.	19.1	0.750	±3	±5	3.10 to 3.91	0.122 to 0.154
¾-in.	15.9	0.625	±3	±5	2.74 to 3.43	0.108 to 0.135
(0.530-in.) ^b	13.4	0.530	±3	±5	2.39 to 3.10	0.094 to 0.122
½-in.	12.7	0.500	±3	±5	2.39 to 3.10	0.094 to 0.122
½-in.	11.1	0.438	±3	±5	2.25 to 2.84	0.088 to 0.112
½-in.	9.52	0.375	±3	±5	2.11 to 2.59	0.083 to 0.102
½-in.	7.93	0.312	±3	±5	1.85 to 2.36	0.073 to 0.093
(0.265-in.) ^b	6.73	0.265	±3	±5	1.60 to 2.11	0.063 to 0.083
¼-in. (No. 3)	6.35	0.250	±3	±5	1.60 to 2.11	0.063 to 0.083
FINE SERIES						
5660 micron (No. 3½)	5.66	0.223	±3	±10	1.28 to 1.90	0.050 to 0.075
4760 micron (No. 4)	4.76	0.187	±3	±10	1.14 to 1.68	0.045 to 0.065
4000 micron (No. 5)	4.00	0.157	±3	±10	1.00 to 1.47	0.039 to 0.055
3360 micron (No. 6)	3.36	0.133	±3	±10	0.87 to 1.22	0.034 to 0.052
2830 micron (No. 7)	2.83	0.111	±3	±10	0.80 to 1.20	0.031 to 0.047
2380 micron (No. 8)	2.38	0.0937	±3	±10	0.74 to 1.10	0.0291 to 0.0433
2000 micron (No. 10)	2.00	0.0787	±3	±10	0.68 to 1.00	0.0268 to 0.0394
1680 micron (No. 12)	1.68	0.0661	±3	±10	0.62 to 0.90	0.0244 to 0.0354
1410 micron (No. 14)	1.41	0.0555	±3	±10	0.56 to 0.80	0.0220 to 0.0315
1190 micron (No. 16)	1.19	0.0469	±3	±10	0.50 to 0.70	0.0197 to 0.0276
1000 micron (No. 18)	1.00	0.0394	±5	±15 ^a	0.43 to 0.62	0.0169 to 0.0244
840 micron (No. 20)	0.84	0.0331	±5	±15 ^a	0.38 to 0.55	0.0150 to 0.0217
710 micron (No. 25)	0.71	0.0280	±5	±15 ^a	0.33 to 0.48	0.0130 to 0.0189
590 micron (No. 30)	0.59	0.0232	±5	±15 ^a	0.29 to 0.42	0.0114 to 0.0165
500 micron (No. 35)	0.50	0.0197	±5	±15 ^a	0.26 to 0.37	0.0102 to 0.0146
420 micron (No. 40)	0.42	0.0165	±5	±25 ^a	0.23 to 0.33	0.0091 to 0.0130
350 micron (No. 45)	0.35	0.0138	±5	±25 ^a	0.20 to 0.29	0.0079 to 0.0114
297 micron (No. 50)	0.297	0.0117	±5	±25 ^a	0.170 to 0.253	0.0067 to 0.0100
250 micron (No. 60)	0.250	0.0098	±5	±25 ^a	0.149 to 0.220	0.0059 to 0.0087
210 micron (No. 70)	0.210	0.0083	±5	±25 ^a	0.130 to 0.187	0.0051 to 0.0074
177 micron (No. 80)	0.177	0.0070	±6	±40 ^a	0.114 to 0.154	0.0045 to 0.0061
149 micron (No. 100)	0.149	0.0059	±6	±40 ^a	0.096 to 0.125	0.0038 to 0.0049
125 micron (No. 120)	0.125	0.0049	±6	±40 ^a	0.079 to 0.103	0.0031 to 0.0041
105 micron (No. 140)	0.105	0.0041	±6	±40 ^a	0.063 to 0.087	0.0025 to 0.0034
88 micron (No. 170)	0.088	0.0035	±6	±40 ^a	0.054 to 0.073	0.0021 to 0.0029
74 micron (No. 200)	0.074	0.0029	±7	±60 ^a	0.045 to 0.061	0.0018 to 0.0024
62 micron (No. 250)	0.062	0.0024	±7	±60 ^a	0.039 to 0.052	0.0015 to 0.0020
53 micron (No. 270)	0.053	0.0021	±7	±90 ^a	0.033 to 0.046	0.0014 to 0.0018
44 micron (No. 325)	0.044	0.0017	±7	±90 ^a	0.031 to 0.040	0.0012 to 0.0016
37 micron (No. 400)	0.037	0.0015	±7	±90 ^a	0.023 to 0.035	0.0009 to 0.0014

^a For sieves from the 1000-micron (No. 18) to the 37-micron (No. 400) size, inclusive, not more than 5 per cent of the openings shall exceed the nominal opening by more than one-half of the permissible variation in maximum opening.

^b The five sieves marked in the first column with the designation *b* may be used instead of the 4-in., 2-in., 1-in., ½-in., and ¼-in. sieves when it is desired to have a series of sieves nesting with the Fine Series and continuing that series with the $\sqrt{2} : 1$ ratio. All of the other sieves listed above are in a $\sqrt{2} : 1$ ratio with the Fine Series within the limit of the specified permissible variations. Care should be taken in designating the five sieves marked with the designation *b*; they should not be designated as 4-in., 2-in., 1-in., ½-in., and ¼-in., but as 4.24-in., 2.12-in., 1.06-in., 0.530-in., and 0.265-in. (or by the manufacturer's nominal values, for example, for the last three 1.050-in., 0.525-in., and 0.263-in.).

The methods which follow have been standardized, and a complete description will be found in the publications cited in the references.

Test 62. Granularmetric Analysis. This is performed by sieving the aggregate through a set of standard sieves of the dimensions given in Table CXLI.⁵⁸

Testing screens of 3-, 2 $\frac{3}{4}$ -, 2 $\frac{1}{2}$ -, 2-, 1 $\frac{1}{2}$ -, 1 $\frac{1}{4}$ -, 1-, $\frac{3}{4}$ -, $\frac{1}{2}$ - and $\frac{1}{4}$ -in. diameter openings and sieves with 10, 20, 30, 40, 50, 80, 100 and 200 meshes per linear inch are commonly used.

For purposes of specification writing and graphical representation of aggregate gradation, mechanical analyses are frequently expressed in one of the following forms:

1. Total per cent smaller than, or passing a given screen or sieve.
2. Total per cent coarser than, or retained on a given screen or sieve.

Control of size and grading of mineral aggregates is of importance from the standpoint of: (1) uniformity; (2) suitability for a given type of construction or use; (3) design of paving mixtures.

For coarser size fragments perforated metal plates with circular openings of specified diameter are used, as well as square mesh sieves of the same nominal side dimensions. The square mesh sieves will pass slightly larger fragments than circular opening screens of the same denomination. The following table shows the sizes of round openings which are approximately equivalent to the stated sizes of square openings.

APPROXIMATELY EQUIVALENT ROUND AND SQUARE OPENING TESTING SCREENS

Square Openings	Round Openings	Square Openings	Round Openings
	In.	In.	In.
No. 8	$\frac{1}{8}$	$1\frac{1}{8}$	$1\frac{1}{2}$
No. 4	$1\frac{1}{8}$	$1\frac{1}{2}$	$1\frac{3}{4}$
$\frac{3}{8}$ in.	$1\frac{3}{8}$	$1\frac{3}{4}$	2
$\frac{1}{2}$ in.	2	2	$2\frac{1}{8}$
$\frac{5}{8}$ in.	$2\frac{1}{8}$	$2\frac{1}{2}$	$2\frac{3}{4}$
$\frac{3}{4}$ in.	$2\frac{3}{8}$	$2\frac{3}{4}$	3
$\frac{7}{8}$ in.	3	3	$3\frac{1}{8}$
1 in.	$1\frac{1}{4}$	$3\frac{1}{2}$	$4\frac{1}{8}$
$1\frac{1}{8}$ in.	$1\frac{3}{8}$	4	$4\frac{1}{4}$

A convenient mechanical sieving apparatus is illustrated in Fig. 361.⁵⁹

The fineness modulus of aggregate is determined by adding the total percentages by weight retained on the following sieves meeting the requirements of Method A.S.T.M. Designation: E 11-39, and dividing by 100: 3-, 1½-, ¾-, ⅜-in., Nos. 4, 8, 16, 30, 50, 100.

(I) *Fine and Coarse Aggregates.* The following method of test has been standardized:⁶⁰

This method of test covers a procedure for the determination of the particle size distribution of fine and coarse aggregates, using sieves with square openings. The method is also applicable to the use of laboratory screens with round openings. It is not intended for use in the sieve analysis of aggregates recovered from bituminous mixtures or for the sieve analysis of mineral fillers.

The apparatus shall consist of the following:

(a) *Balance:* The balance or scale shall be sensitive to within 0.1 per cent of the weight of the sample to be tested.

(b) *Sieves:* The sieves with square openings shall be mounted on substantial frames constructed in a manner that will prevent loss of material during sieving. Suitable sieve sizes shall be selected to furnish the information required by the specifications covering the material to be tested. The woven wire cloth sieves shall conform to A.S.T.M. Designation: E 11.

NOTE.—If round-hole perforated plate screens are used, the openings shall conform to the applicable dimensions and tolerances prescribed in A.S.T.M. Designation: E 11.



Courtesy of Howard & Morse.

FIG. 361.—Mechanical Sieving Apparatus.

Samples for sieve analysis shall be obtained, by quartering or by use of a sampler, from a representative sample selected from the material to be tested.

Samples of fine aggregate for sieve analysis shall weigh, after drying, not less than the amount indicated in the following table:

Material with at least 95 per cent finer than a No. 10 (2000-micron) sieve	100 g.
Material with at least 90 per cent finer than a No. 4 (4760-micron) sieve and more than 5 per cent coarser than a No. 10 (2000-micron) sieve	500 g.

Samples of coarse aggregate for sieve analysis shall weigh, after drying, not less than an amount indicated in the following table:

Nominal Maximum Size of Particle, in.	Minimum Weight of Sample, g. ^a
$\frac{3}{8}$	1,000
$\frac{1}{2}$	2,500
$\frac{3}{4}$	5,000
1	10,000
1 $\frac{1}{2}$	15,000
2	20,000
2 $\frac{1}{2}$	25,000
3	30,000
3 $\frac{1}{2}$	35,000

^a For samples weighing 5000 g. or more it is recommended that sieves mounted in frames 16 in. in diameter or larger be used.

In the case of mixtures of fine and coarse aggregates, the material shall be separated into two sizes on the No. 4 (4760-micron) sieve and the samples of fine and coarse aggregates shall be prepared as described above.

In the case of fine aggregate, the material finer than the No. 200 (74-micron) sieve shall be determined in accordance with A.S.T.M. Designation: C 117 and the sieve analysis made on the material coarser than the No. 200 (74-micron) sieve.

Samples shall be dried to substantially constant weight at a temperature not exceeding 110° C. (230° F.).

The sample shall be separated into a series of sizes using such sieves as are necessary to determine compliance with the specifications for the material under test. The sieving operation shall be conducted by means of a lateral and vertical motion of the sieve, accompanied by jarring action so as to keep the sample moving continuously over the surface of the sieve. In no case shall fragments in the sample be turned or manipulated through the sieve by hand. Sieving shall be continued until not more than 1 per cent

by weight of the residue passes any sieve during 1 min. On that portion of the sample retained on the No. 4 (4760-micron) sieve, the above described procedure for determining thoroughness of sieving shall be carried out with a single layer of material. When mechanical sieving is used, the thoroughness of sieving shall be tested by using the hand method of sieving as described above.

The weight of each size shall be determined on a scale or balance conforming to the requirements specified.

The results of the sieve analysis shall be reported as follows: (a) total percentages passing each sieve, or (b) total percentages retained on each sieve, or (c) percentages retained between consecutive sieves, depending upon the form of the specifications for the use of the material under test. Percentages shall be reported to the nearest whole number and shall be calculated on the basis of the weight of the test sample including any material finer than the No. 200 (74-micron) sieve.

(II) *Coarse Particles in Mixtures of Asphalt with Mineral Matter.* The following method of test has been standardized:²¹

This method of test covers the procedure for determining the amount of particles of mineral or other insoluble matter in asphaltic mixtures that are retained upon a No. 200 (74-micron) sieve.

The material shall be sampled in accordance with A.S.T.M. Designation: D 140.

The sample as received shall be completely melted in an oven at the lowest possible temperature, stirred well to insure homogeneity, and samples for test taken immediately.

The apparatus shall consist either of a 3-in. No. 200 (74-micron) sieve or a crucible sieve, 1¾ in. in diameter at the top and 1¼ in. in diameter at the bottom containing a No. 200 (74-micron) sieve. The sieve shall conform to A.S.T.M. Designation: E 11.

A 3-in. No. 200 (74-micron) sieve for comparison purposes shall be retained in the laboratory as a standard. Whenever a new sieve is secured, a practical test of its accuracy should be made by running on it, and on the standard sieve, a comparison test, using powdered mineral matter that has a considerable percentage of coarse particles. A reserve stock of such powdered mineral filler should be kept for this purpose.

The sieve shall be dried in an oven at 105 to 110° C., cooled,

and then weighed on an analytical balance, the weight being recorded to the nearest 1 mg.

A sample of the asphalt sufficient to yield about 1 g. of mineral matter shall be weighed into a 400-ml. beaker. Then carbon disulfide, carbon tetrachloride, or benzol shall be added and the mixture warmed until the asphalt is entirely dissolved. The solution obtained shall be poured through the weighed sieve, and the residue in the beaker shall be washed onto the sieve, using the solvent employed.

The sieve containing the residue shall be washed with a fine jet of the solvent from a wash bottle until free from asphalt and fine mineral matter, then dried, tapped lightly to remove any fine mineral matter held on the wires, and weighed. During the washing, the sieve shall be held so that the liquid will drain through readily.

The percentage of coarse particles in the mineral matter shall be calculated from the weight of the sample taken, the percentage of matter insoluble in carbon disulfide, and the weight of the residue retained on the sieve.

(III) *Mineral Filler.* The following method of test has been standardized:⁶²

This method of test covers the sieve analysis of mineral fillers used in road and paving materials.

(a) Balance: The balance shall be sensitive to 0.05 g.

(b) Sieves: The sieves shall conform to A.S.T.M. Designation: E 11 and shall include the No. 200 (74-micron) sieve and such other sieves as may be required by the specifications for the mineral filler.

NOTE.—In general specifications require the use of the No. 200 (74-micron), No. 80 (177-micron), and No. 30 (590-micron) sieves.

The sample for sieve analysis shall be obtained by quartering, or by use of a sampler, from a representative sample selected from the material to be tested. Approximately 50 g. of dry material is required for each test.

The sample shall be dried to substantially constant weight at a temperature not exceeding 110° C. (230° F.).

The 50.0 ± 5.0 g. sample of the dried mineral filler shall be weighed to the nearest 0.1 g. and placed on the No. 200 (74-micron) sieve which shall be thoroughly clean and dry. The sieve,

with pan and cover attached, shall be held in one hand in a slightly inclined position so that the sample will be well distributed over the sieve, at the same time gently striking the side about 150 times per minute against the palm of the other hand on the up stroke. The sieve shall be turned every 25 strokes about one-sixth of a revolution in the same direction. The operation shall be continued until not more than 0.05 g. passes through the sieve in 1 min. of continuous sieving.

The portion of the sample retained on the sieve shall then be weighed to the nearest 0.1 g., and placed on the sieve with the next larger opening for the series selected for the sieve analysis. Sieving shall be continued in a similar manner, using successively each of the selected series of sieves in the order of increasing size of opening, and recording the weight of that portion of the sample retained on each sieve. The weight of material and percentage of the sample passing each of the sieves shall be calculated.

Washers, shot, or slugs shall not be used on the sieves.

Mechanical sieving devices may be used, but the filler shall not be rejected if it meets the requirements when tested by the hand method described. When mechanical sieving devices are used their thoroughness of sieving shall be tested by using the hand method.

The report shall include the following:

- (a) Results of the sieve analysis reported as the total percentage passing each sieve, expressed to the nearest 0.5 per cent, and
- (b) The method of sieving used.

The percentages obtained by the same operator in duplicate tests on portions of the same sample should not differ by more than one passing any one sieve. The percentages obtained by different operators in different laboratories should not differ by more than two passing any one sieve.

(IV) *Material Finer Than No. 200 Mesh Sieve.* The following method of test has been standardized: ⁶⁸

This method of test outlines the procedure for determining the total quantity of material finer than a standard No. 200 (74-micron) sieve in aggregates.

The apparatus shall consist of the following:

- (a) Sieves: A nest of two sieves, the first being a No. 200 (74-micron) sieve conforming to the requirements of A.S.T.M.

Designation: E 11 and the second, a sieve having approximately 16 meshes per linear inch.

(b) Container: A pan or vessel of a size sufficient to contain the sample covered with water and to permit of vigorous agitation without inadvertent loss of any part of the sample or water.

The test sample shall be selected from material which has been thoroughly mixed and which contains sufficient moisture to prevent segregation. A representative sample, sufficient to yield not less than the appropriate weight of dried material, as shown in the following table, shall be selected:

Nominal Diameter of Largest Particle, In.	Approximate Minimum Weight of Sample, Kg.
$\frac{1}{2}$	0.5
$\frac{3}{4}$	2.5
$1\frac{1}{2}$ or over.....	5.0

The test sample shall be dried to constant weight at a temperature not exceeding 110° C. (230° F.) and weighed to the nearest 0.02 per cent.

The test sample after being dried and weighed shall be placed in the container and sufficient water added to cover it. The contents of the container shall be agitated vigorously and the wash water poured immediately over the nested sieves, arranged with the coarser sieve on top.

The agitation should be sufficiently vigorous to result in the complete separation from the coarse particles of all particles finer than the No. 200 (74-micron) sieve and bring the fine material into suspension, in order that it will be removed by decantation of the wash water. Care shall be taken to avoid, as much as possible, the decantation of the coarse particles of the sample. The operation shall be repeated until the wash water is clear.

All material retained on the nested sieves shall be returned to the washed sample. The washed aggregate shall be dried to constant weight at a temperature not exceeding 110° C. (230° F.) and weighed to the nearest 0.02 per cent.

The results shall be calculated from the following formula:

$$\left. \begin{array}{l} \text{Percentage of material finer} \\ \text{than No. 200 sieve} \end{array} \right\} = \frac{\text{Orig. dry wt.} - \text{Dry wt. after washing}}{\text{Orig. dry wt.}} \times 100$$

When check determinations are desired, the wash water shall be either evaporated to dryness or filtered through tared filter paper which shall subsequently be dried, the residue weighed, and the percentage calculated from the following formula:

$$\left. \begin{array}{l} \text{Percentage of material finer} \\ \text{than No. 200 sieve} \end{array} \right\} = \frac{\text{Wt. of residue}}{\text{Orig. dry wt.}} \times 100$$

(V) *Size Distribution of Microscopic Particles.* Various procedures have been suggested for this purpose, including a standard test for fineness of portland cement,⁶⁴ a method of evaluating fine fillers,⁶⁵ a microscopic examination,⁶⁶ etc. The following method of test has been standardized:⁶⁷

This method of test covers the procedure for determining the particle size of particulate substances in absolute units, as far as the particle shape will permit. It is intended to cover the range of sizes between the 74-micron (No. 200) sieve and 0.2 micron. The method is applicable in its entirety to homogeneous materials. In the case of mixtures, the extent of application is limited by the properties of the components of the mixture.

Dispersion: In microscopy it refers to the distribution of the individual particles. In statistical work it is measured by the standard deviation.

NOTE.—Both uses of the word seem too generally accepted to warrant a change in either.

Individual Particle: Those minute units of matter (of which the material is composed) whose size and shape depend only on the force of cohesion. Such particles cannot be subdivided without separating like molecules that are within the range of the cohesive attraction of one another.⁶⁸

Aggregate: A group of two or more attached particles.

NOTE.—There are three forms of aggregates: namely, ultimate working unit, flocculate, and apparent flocculate.

Ultimate Working Unit: An individual particle or any group of individual particles that are so firmly held together by forces of adhesion that they remain intact as a group throughout the duration of their application.

Flocculate: Flocculation occurs only where particles have been incorporated in a liquid or plastic medium. A flocculate is a group

of particles held together by a force, apparently arising from interfacial tension. This force of flocculation is relatively weak, consequently flocculates do not function as large individual particles, and cannot be classified under ultimate working units. Under slight continuously applied force, flocculates are constantly breaking down and reforming.

Apparent flocculates; as found in gas-floated or air-floated particles show a somewhat similar type of aggregation. Usually the force of attraction between the particles is relatively weak and the particles can readily be dispersed in suitable media. For practical purposes, the more inclusive term, aggregate, is preferable to flocculate for gas-floated or air-floated particles.

Average Diameter: The diameter of a hypothetical particle which, in some particular way, represents the total mass of particles.

Non-uniformity: A non-uniform particulate substance is one in which the constituent particles differ from one another in diameters.

Rough Separation into Size Groups: For pigments or other materials which are essentially less than 6 microns proceed directly with the preparation of the mount.

For wide ranges of distribution of particle size in sub-sieve size portions, auxiliary separation into groups of sizes is necessary to facilitate measurement with the microscope. Rough separation is permissible as exact size measurement in microns follows. The following general ranges of particle diameter are proposed for the separation:

A	24 microns and larger
B	24 to 12 microns
C	12 to 6 microns
D	6 to 0.2 microns

For specific purposes A or D may be further separated.

NOTE.—If the sample contains particles larger than the 74-micron (No. 200) sieve, the material shall be separated by sieving wet in a suitable fluid. The portion which passes the sieve may be recovered by evaporation of the fluid. The portion which remains on the sieve may be subjected when dry to a standard sieve test and additional material that passes the 74-micron (No. 200) sieve added to that which is to be tested.

Separation shall be made by air or liquid elutriation or by sedimentation at room temperature. The volume of liquid in the elutriator or sedimentation apparatus should be between 20 and 100

ml. per gram of solid. With finer particles and with material of lesser density, the greater volumes should be used. The technique involved in the elutriation or settling is necessarily influenced by the type of material being measured and the dispersing liquids. The size of the vessel and dispersion will be specified in terms of the uniformity obtained. The microscope measurement will be used as a check upon the effectiveness of the separation. For accurate results at least 50 per cent by weight must fall within the boundaries of the suggested ranges set for the elutriation. Of the material falling outside the boundaries, not over 5 per cent by weight may fall outside of a range which is one half of the lower limit and twice the upper limit of the range under consideration.

The fluid used in the elutriation or sedimentation shall contain no dissolved solids which would influence the weight determination of a given portion, that is, the fluid shall be 100 per cent volatile. It shall not react chemically with the material being elutriated. Water, kerosine, alcohols, and the like may be used. It is suggested that the filtering of any elutriator portion should be through alundum to avoid contamination of the sample with paper or pulp fibers. As an alternative, the solid may be recovered from each stage of the elutriator by evaporation of a concentrated slurry, providing the temperatures used for the evaporation do not alter the specific product. The total weight of the portion recovered shall check within 5 per cent the weight of material initially placed in the elutriator.

All limits are specified to insure good microscopic technique. Good elutriation technique for analytical purposes falls well within these limits.

NOTE.—Quite obviously, materials containing components of different densities will elutriate or settle in ranges of low uniformity. The present requirements can only be applied when the material meets the following requirements for homogeneity. If the materials can be readily identified under the microscope, they may be measured individually and the results considered as exact, notwithstanding wide differences in density between two materials which may cause lack of uniformity. In other cases, such as cement or coal, where the presence respectively of gypsum and ash constituents may affect the test, these effects may be considered negligible for the normal type of products.

Preparation of the Mount: Any one of a number of methods of preparing the mount may be used, the criterion of suitability being conformity to the following requirements:

The particles shall be essentially in one plane.

The particles shall be free from motion.

The particles shall be dispersed, showing individual grains instead of aggregates and flocculates.

The particles shall not be ground in mounting.

The mount shall be truly representative of the distribution of sizes in the material.

The mounting medium shall be selected to give maximum definition.

Fine Materials, Pigments, etc.: The type of mount that should be used depends on the physical properties of the particles, and for this reason no definite requirements are specified. The general procedure shall be to place the material being tested on a microscope slide, rub it out in a solution of gum or resin in a solvent until the particles are well dispersed. After the solvent has evaporated, the non-volatile constituent of the dispersing agent serves to cement the particles to the slide in the dispersed state.

NOTE.—Detailed technique will be found in the work of Dunn⁶⁹ and Green.⁷⁰ The former uses balsam in xylol, and the latter turpentine and dammar. Other mounting media suggested are 0.5 per cent glycerin in alcohol, rubber cement, styrax in xylol, and the chlorinated naphthalenes and saponin.

Coarse Material, Ground Products, etc.: In the case of coarse materials it is often advantageous to use the so-called dry mount, in which the dispersing agent used is volatile and can be driven off after the material has been dispersed on the slide. The uniformity of the mount when examined microscopically is the best criterion of an acceptable mount.

(I) *Direct Observation Method.* Direct observation using the Filar micrometer⁷¹ has been used with fair success and reasonable degree of accuracy on the coarser fraction of particulate substances. The Filar micrometer is an attachment which fits into the draw tube of the microscope and is fitted with a Ramsden-type eyepiece which permits focusing on a movable cross hair activated by a micrometer screw. This instrument permits direct measurement of the particle as viewed on the slide. This method, however, is not recommended for materials in the 0.2 to 6 micron class size.

(II) *Projection Method.* The projection method which involves throwing the image of the particle on a screen has several distinct advantages. It further magnifies the image, permits focus-

ing through the depth of the mount insuring a proper focus and count of all the particles.

(III) *Photomicrographic Method.* The photomicrographic method⁷² which involves taking a photomicrograph of the particles and subsequently measuring them on a print or by projecting the

TABLE CL
DIAMETER RANGE IN MICRONS

General Case (20 Per Cent or More Under 6 Microns)	Special Case (Less than 20 Per Cent Under 6 Microns)
72 to 60	
60 to 48	
48 to 36	
36 to 24	
24 to 18	
18 to 12	
12 to 9	
9 to 6	
6.00 to 5.00	Same Scale and
5.00 to 4.00	6 to 4.5
4.0 to 3.5	4.5 to 3
3.5 to 3.0	3 to 1.5
3.0 to 2.5	1.5 to 0
2.5 to 2.0	
2.0 to 1.8	
1.8 to 6	
1.6 to 4	
1.4 to 2	
1.2 to 0	
1.0 to 9	
0.9 to 8	
0.8 to 7	
0.7 to 6	
0.6 to 5	
0.5 to 4	
0.4 to 0.3	
0.3 to 0.2	
0.2	

negative on the screen is also recommended. This method has the very decided advantage of making a permanent record of the sample and permits eye comparisons of two or more samples. In the use of this method it is extremely important that the mount be made according to the method outlined under the method of Green and fulfill the requirements specified there.

Measurement of Diameter: In each of the three methods referred to in the preceding sections, the horizontal diameter shall be measured, unless the particles are very uniform in shape and in that event a diameter which most nearly represents the average

shall be selected. For irregularly shaped particles, the horizontal diameter splitting the particle in half in one direction consistently maintained has been shown^{7a} to give fairly consistent and accurate results. For flat plates it is necessary to consider the third dimension for accurate results. In any case, in reporting results, the diameter measured shall be specified.

Scale Limits for Measurement: If more than 10 per cent by weight is coarser than 6.00 microns, the sample shall be elutriated prior to microscopic measurement. A graded scale for microscopic sizing is given in Table CI.

Two hundred and fifty particles shall be measured in each of three fields, taken at random.

The magnification shall be dependent on the class size being measured, and shall be sufficient to permit the differentiation specified above.

Expression of Results: Results shall be expressed using one or more of the following average diameters:

Average Diameter	To Be Used:	Formula
d_1	As arithmetical mean.....	$d_1 = \frac{\sum nd}{\sum n}$
d_2	To determine S when ρ is known.....	$d_2 = \sqrt{\frac{\sum nd^2}{\sum n}}$
d_4	To determine volume or weight distribution.....	$d_4 = \sqrt[4]{\frac{\sum nd^4}{\sum n}}$
D	To determine N when ρ is known.....	$D = \sqrt[3]{\frac{\sum nd^3}{\sum n}}$
Δ	To determine S when N is known.....	$\Delta = \sqrt{\frac{\sum nd^2}{\sum n}}$
M_g	As geometric mean or median.....	$\log M_g = \frac{\sum n \log d}{\sum n}$

Symbols

- S_m = specific surface in square meters per gram
 S_p = specific surface in square meters per milliliter
 N_m = number of particles per gram
 N_p = number of particles per milliliter
 Σn = number of particles in any given mass
 ρ = density of the material
 θ = arithmetic standard deviation
 θ_g = geometric standard deviation
 P.E. = probable error.

If 90 per cent by weight fall in any one class size, a frequency curve shall be given.

In the 0 to 6-micron class, either a frequency curve or the distribution by weight shall be given.

In elutriated products, weight distribution shall be expressed as follows:

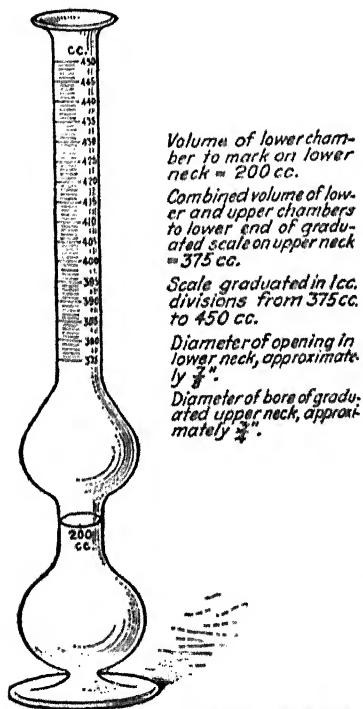
Weight distribution shall be the basis of comparison for all elutriated samples. The weight distribution for each elutriated portion shall be calculated on the basis of spherical particles using the arithmetic mean of the microscopic class interval as the average diameter for all particles in that range. Each of the elutriated fractions shall total in weight distribution to the percentage retained in the elutriator and the composite curve shall be a summation of the distribution values in each range. The degree of overlapping is a measure of the efficiency of the elutriation and it shall not be abnormal in amount.

Test 63. Adsorptive Capacity of Fine Fillers. Most fillers exert more or less of an adsorptive affinity in respect to bituminous substances, whereby a small proportion of the latter remains in combination with the mineral filler upon extraction with solvents. The following test has been proposed as a measure of this "adsorptive capacity": Weigh 5 g. of the filler into a flask and add 30 ml. of a 0.5 per cent solution of a standard asphalt dissolved in C.P. benzol (it is recommended that a 50 to 60 penetration asphalt cement of known origin be used for the purpose). Agitate for $\frac{1}{2}$ hour and filter through paper. Compare the color of the filtrate with a standard series prepared from the same asphalt dissolved in benzol in varying percentages (i.e., 0.1, 0.2, 0.3, 0.4 and 0.5 per cent solutions). The diminution of color is a measure of the adsorptive capacity of the filler.

It has been observed that the addition of mineral fillers often causes the removal of asphaltic resins from the bituminous binder by adsorption, and tends to reduce the viscosity of the mixture.⁷⁴

A "kerosine absorption test" has been proposed for gauging the capacity of aggregates to carry bituminous binders, in which 100 g. of the dry aggregate is saturated with kerosine and centrifuged for 2 minutes at a force of 400 times its gravity. The weight of kerosine retained, expressed as a percentage of the dry aggre-

gate, is termed the "centrifuge kerosine equivalent" and constitutes a measure of the optimum amount of liquid asphalt to be incorporated into the mixture.⁷⁵



Volume of lower chamber to mark on lower neck = 200 cc.

Combined volume of lower and upper chambers to lower end of graduated scale on upper neck = 375 cc.

Scale graduated in 10 cc. divisions from 375 cc. to 450 cc.

Diameter of opening in lower neck, approximately $\frac{1}{4}$ in.

Diameter of bore of graduated upper neck, approximately $\frac{3}{4}$ in.

Courtesy A.S.T.M.

FIG. 362.—Graduated Flask for Testing Fine Aggregate.

Test 64. Specific Gravity. Several methods have been standardized⁷⁶ for ascertaining the apparent specific gravity of mineral aggregates, fine mineral fillers, pigments, etc. The following procedure⁷⁷ has been standardized for finding the apparent specific gravity of fine aggregates:

Method (1): This test is intended for use in making determinations of bulk and apparent specific gravity, and absorption (after 24 hr. in water at room temperature) of fine aggregate. The bulk specific gravity is the value generally desired for calculations in connection with portland-cement concrete.

This method determines directly the bulk specific gravity, or the bulk specific gravity on the basis of weight of saturated surface-dry aggregate, or the apparent specific gravity.

The apparatus shall consist of the following:

- (a) **Balance:** A balance having a capacity of 1 kg. or more and sensitive to 0.1 g. or less.
- (b) **Flask:** A volumetric flask of 500-ml. capacity, calibrated to 0.1 ml. at 20° C. (Fig. 362).
- (c) **Conical Mold:** A conical metal mold, 1½ in. in diameter at the top, 3½ in. in diameter at the bottom, and 2¾ in. in height.
- (d) **Tamping Rod:** A metal tamping rod, weighing 12 oz., and having a flat circular tamping face 1 in. in diameter.

Approximately 1000 g. of the fine aggregate selected from the sample by the method of quartering shall be spread on a flat

NOTE.—Where the absorption and specific gravity values may be utilized as a basis for designing concrete mixtures with aggregates normally used in a moist condition, the requirement of drying to constant weight may be eliminated.

surface, exposed to a gently moving current of warm air, and stirred frequently to secure uniform drying. This operation shall be continued until the sand approaches a free flowing condition. The fine aggregate shall then be placed loosely in the conical mold, the surface lightly tamped 25 times with the metal rod, and the mold lifted vertically. If free moisture is present, the cone of fine aggregate will retain its shape. Drying with constant stirring shall be continued and tests made at frequent intervals, until the cone of fine aggregate slumps upon removal of the mold. This indicates that the fine aggregate has reached a surface-dry condition.

NOTE.—The procedure described above is intended to insure that the first trial determination shall be made with some free water in the sample. If the cone of sand slumps on the first trial, the sand has been dried past the saturated and surface-dry condition. In this case a few milliliters of water shall be thoroughly mixed with the sand and the sample permitted to stand in a covered container for 30 min. The process of drying and testing the sand shall then be resumed.

A 500.0-g. sample of the material prepared as described above shall be introduced immediately into the flask and the flask filled almost to the 500-ml. mark with water at a temperature of 20° C. The flask shall then be rolled on a flat surface to eliminate all air bubbles, after which it shall be placed in a constant temperature bath maintained at 20° C. After approximately 1 hr. it shall be filled with water to the 500-ml. mark and the total weight of water (see Note) introduced into the flask shall be determined to the nearest 0.1 g.

NOTE.—If desired, the quantity of water necessary to fill the flask may be determined volumetrically by the use of a burette accurate to 0.1 ml.

The fine aggregate shall be removed from the flask and dried to constant weight at a temperature of 100 to 110° C., cooled to room temperature in a desiccator, and weighed.

The bulk specific gravity shall be calculated from the following formula:

$$\text{Bulk specific gravity} = \frac{A}{V - W}$$

where A = weight in grams of oven-dry sample in air,

V = volume in milliliters of flask, and

W = weight in grams or volume in milliliters of water added to flask.

The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk specific gravity (saturated surface-dry basis)} = \frac{500}{V - W}$$

The apparent specific gravity shall be calculated from the following formula:

$$\text{Apparent specific gravity} = \frac{A}{(V - W) - (500 - A)}$$

The percentage of absorption shall be calculated from the following formula:

$$\text{Percentage of absorption} = \frac{500 - A}{A} \times 100$$

Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

Method (II): The following procedure⁷⁸ is adapted for finding the specific gravity and absorption of coarse aggregates:

This method of test is intended for use in making determinations of bulk and apparent specific gravity, and absorption (after 24 hr. in water at room temperature) of coarse aggregate.

This method determines directly the bulk specific gravity or the bulk specific gravity on the basis of weight of saturated surface-dry aggregate, or the apparent specific gravity.

The apparatus shall consist of the following:

(a) Balance: A balance having a capacity of 5 kg. or more and sensitive to 0.5 g. or less.

(b) Wire Basket: A wire basket of No. 4 mesh wire, approximately 8 in. in diameter and 8 in. in height.

(c) Suitable Container for immersing wire basket in water and suitable apparatus for suspending wire basket from center of scale pan of balance.

After thoroughly washing to remove dust or other coatings from the surface of the particles, the sample shall be dried to constant weight at a temperature of 100 to 110° C. (Note) and then immersed in water at 15 to 25° C., for a period of 24 hr.

NOTE.—Where the absorption and specific gravity values may be utilized as a basis for designing concrete mixtures with aggregates normally used in a moist condition, the requirement of drying to constant weight may be eliminated.

It shall then be removed from the water and rolled in a large absorbent cloth until all visible films of water are removed, although the surfaces of the particles still appear to be damp. The larger fragments may be individually wiped. Care should be taken to avoid evaporation during the operation of surface drying. The weight of the sample in the saturated surface-dry condition shall then be obtained. This and all subsequent weights shall be determined to the nearest 0.5 g.

After weighing, the saturated surface-dry sample shall be placed immediately in the wire basket and its weight in water determined. The sample shall then be dried to constant weight at a temperature of 100 to 110° C., cooled to room temperature, and weighed.

The bulk specific gravity shall be calculated from the following formula:

$$\text{Bulk specific gravity} = \frac{A}{B - C}$$

where A = weight in grams of oven-dry sample in air,

B = weight in grams of saturated surface-dry sample in air,

and C = weight in grams of saturated sample in water.

The bulk specific gravity on the basis of weight of saturated surface-dry aggregate shall be calculated from the following formula:

$$\text{Bulk specific gravity (saturated surface-dry basis)} = \frac{B}{B}$$

The apparent specific gravity shall be calculated from the following formula:

$$\text{Apparent specific gravity} = \frac{A}{A - C}$$

The percentage of absorption shall be calculated from the following formula:

$$\text{Percentage of absorption} = \frac{B - A}{A} \times 100$$

Duplicate determinations should check to within 0.02 in the case of specific gravity and 0.05 per cent in the case of percentage of absorption.

The percentage of voids in aggregates (see also Test 7f) may readily be calculated from the specific gravity by the following formula:⁷⁹

$$\text{Percentage of voids} = \frac{(\text{Sp. gr.} \times 62.355) - \text{Wt.}}{\text{Sp. gr.} \times 62.355} \times 100$$

where Sp. gr. = the bulk specific gravity of the aggregate as determined by the appropriate method,

62.355 = the weight in pounds of 1 cu. ft. of water at the standard temperature of 16.7° C. (62° F.), and

Wt. = the weight in pounds per cubic foot of the aggregate.

Method (III): The following method of test has been standardized for determining the surface moisture in fine aggregates:⁸⁰

This method of test covers the procedure for determining the approximate percentage of surface moisture in fine aggregate. The range of the apparatus is between the specific gravities of 2.2 for aggregate containing 10 per cent of moisture and 2.85 for dry aggregate.

This method determines only surface moisture, that is, moisture on the outside of the particles. The moisture absorbed within the particles does not add to the volume of the particles and, therefore, does not make itself evident in this test.

The apparatus shall consist of the following:

(a) Balance: A balance, preferably of the torsion type, having a capacity of 2 kg. or more and sensitive to 0.5 g. or less.

(b) Flask: A special graduated flask of the type, and conforming to the dimensions, shown in Fig. 362.

A sample weighing 1 kg. shall be selected which shall be as truly representative of the fine aggregate as possible. It shall be well mixed and 500 g. shall be immediately weighed out, permitting moisture to evaporate as little as possible from the sample.

The graduated flask shall be filled to the 200-ml. mark on the lower neck with water at room temperature. The 500-g. sample

of damp aggregate shall then be slowly poured into the flask, and the contents of the flask agitated or stirred to free any entrained air bubbles. The combined volume, in milliliters, of the water and fine aggregate shall be read on the scale on the upper neck of the flask. This method requires that the bulk specific gravity of the saturated surface-dried fine aggregate, determined in accordance with A.S.T.M. Designation: E 12.

The percentage of surface moisture in the fine aggregate (air-dry basis) shall be calculated from the formula:

$$M = \frac{V - \frac{500}{\text{Sp. gr.}} - 200}{200 + 500 - V} \times 100$$

where M = approximate percentage of surface moisture,

V = combined volume in milliliters of the water and fine aggregate in the flask, and

Sp. gr. = bulk specific gravity of the saturated surface-dried fine aggregate.

Duplicate determinations should check within 0.5 per cent.

NOTE.—Variations in the composition of the rock grains of the fine aggregate which result in variations of 0.05 in the approximate apparent specific gravity of the aggregate will cause inaccuracy in results equal to from 1 to 1.5 per cent of moisture.

Organic Particles, Fibers, Fillers, etc.:

These constituents may be examined microscopically, or by any of the conventional methods, the scope of which fall outside of the present publication.

CHAPTER XXXIV

EXAMINATION OF BITUMINIZED FABRICS

This caption includes the following groups of products, viz.:

- Q—Prepared roofings.
- R—Composition shingles.
- S—Deck and porch coverings.
- T—Bituminized fabrics for constructing built-up roofs.
- U—Bituminized fabrics for constructing waterproofing membranes.
- V—Electrical insulating tape.
- W—Waterproof papers for wrapping and packing.
- X—Waterproof papers for insulating against heat or cold.
- Y—Felt-base floor coverings (surfaced with linseed oil and pigment composition).
- Z—Expansion joints for pavements.

These are constructed as shown in Table CII, where the index *a* indicates that asphaltic compositions have been used, and *t* signifies that coal tar (pitch) *et al.*, have been used.

Since the finished products falling in this class are constructed in many different ways, it will obviously be impracticable to describe in detail the analytical methods applicable to each. The ones which follow have been devised specifically for examining prepared roofings,¹ but with these as a starting-point, others may readily be evolved for testing floor coverings, waterproof membranes, sheathing and insulating papers, etc.

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT

Test 65. Weight per Unit Area. The following methods have been standardized:

(I) *For Saturated Felted and Woven Fabrics:*² The width of each roll taken shall be measured to the nearest $\frac{1}{32}$ in. The minimum and maximum width so obtained shall be reported.

TABLE CLI
STRUCTURE OF BITUMINIZED FABRICS

	Paper		Burlap		Duck		Light Cotton Fabric		Rag-Felt		Asbestos Felt		Burlap and Rag or Asbestos Felt		Paper and Light Cotton Fabric	
	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>	<i>a</i>	<i>t</i>
<i>Single Layered:</i>																
Saturated only.....	WX	WX	U	U	TUY	TU	T
Coated only (one or two sides).....	W	U	U	S
Saturated and coated.....	X	U	U	QS	V	QR	Q
<i>Laminated (Bituminated):</i>																
Layers saturated only.....	Z	Z	Q	QT	UZ	U
Layers saturated and coated.....	Q	Q	Q
Layers unsaturated	W	W	W	W
One layer unsaturated and others saturated.....	W	W	W	W

NOTE. Letters in heavy type indicate the more important groups of products.

Each roll taken shall be weighed intact to the nearest $\frac{1}{4}$ lb. and the minimum and maximum gross weight shall be reported.

Each roll selected shall be stripped. All the wrappers and packing material shall be weighed together to the nearest $\frac{1}{4}$ lb. and the average weight thereof per roll shall be reported. Each roll shall then be unwound, observing the workmanship and finish while so doing. The length and width of each roll shall thereupon be measured to the nearest $\frac{1}{4}$ in. and the square feet of material contained in each roll calculated.

The rolls shall be rewound, fastened with pieces of light string and then each roll shall be weighed to the nearest $\frac{1}{4}$ lb. In the case of felted fabrics, the weight of each roll in pounds per 100 sq. ft. shall be calculated and the maximum and minimum weights reported. The average weight for the rolls sampled, which shall

be regarded as the corresponding weights of the entire lot, shall also be reported. In the case of woven fabrics, the weight of each roll shall be calculated in ounces per square yard, and the minimum weight reported, likewise recording the average for the rolls sampled, which shall be regarded as the corresponding weights of the entire lot.

From the rolls examined, the one whose weight per unit area is nearest the average weight per unit area of the lot shall be selected. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge the sheet shall be cleanly cut across at right angles to the edges. A sample measuring 30 in. in the direction of the roll's length shall be removed. If the material is surfaced with sand or other fine material, any detached particles belonging to each 30-in. section shall be retained. The width of each section shall be measured to the nearest $\frac{1}{32}$ in. Each section, together with any detached surfacing, shall be weighed to the nearest gram. The weight in pounds per 100 sq. ft. or the weight in ounces per sq. yd. shall be calculated from the formulas:

$$\text{Pounds per 100 sq. ft.} = 1.0582 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

$$\text{Ounces per sq. yd.} = 1.5238 \times \frac{\text{Weight of 30-in. section in grams}}{\text{Width of 30-in. section in inches}}$$

The weight so determined shall check within 1 per cent of the average weight per unit area of the lot. If the sample fails to do this, then additional samples shall be cut from the same roll, until one is obtained which does, and this sample shall be reserved for further examination as described.

NOTE.—As a referee method or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll sampled as above.

If the material is surfaced with sand or other finely comminuted material, the surfacing shall be swept with a moderately stiff brush from all the 30-in. samples taken. All the material thus removed shall be caught and weighed to the nearest ounce. From the aggregate areas of all the sections taken, the average weight of de-

From the above, the net weight, exclusive of loose surfacing, of roofing per 108 sq. ft. contained in each roll selected shall be calculated. The minimum weight per 108 sq. ft. shall be recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

The average weight per 108 sq. ft. for the rolls examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the lot. (1a)

From the rolls examined, the one whose weight per 108 sq. ft. is nearest the average weight of the lot shall be selected. The roll so selected shall be laid flat, the first convolution or two carefully unwound, and with a knife and straight edge, the sheet shall be cleanly cut across at right angles to the edges. A sample measuring 30 in. ($\pm \frac{1}{32}$ in.) in the direction of the roll's length shall then be removed, and the width of the surfaced area shall be measured to the nearest $\frac{1}{32}$ in. The weight of the sample in ounces shall be determined, neglecting any loose surfacing. The weight in pounds per 108 sq. ft. shall then be calculated as follows:

$$\left. \begin{array}{l} \text{Pounds per} \\ 108 \text{ sq. ft.} \end{array} \right\} = \frac{\text{Weight of 30-in. section in ounces}}{\text{Width of 30-in. section in inches}} \times 32.4 \text{ (1b)}$$

The weight so determined shall check within 1.5 per cent of the average weight per 108 sq. ft. of the shipment (1a). If the sample fails to conform to this requirement, then additional samples shall be cut from the same roll, until one is obtained which does, which sample shall be reserved for further examination.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, a 30-in. sample shall be taken and examined separately from each roll selected.

(III) *For Mineral-surfaced Shingles:*^a Each bundle selected shall be weighed to the nearest $\frac{1}{4}$ lb. The packing material shall be weighed to the nearest $\frac{1}{4}$ lb. and the weight recorded separately for each bundle. The average weight of packing material per "square" shall be recorded.

The shingles in each bundle, "a," shall be counted and the workmanship and finish of the shingles shall be observed. The weight of any loose surfacing that may fall off during this operation shall be recorded.

From the above the net weight of each bundle, exclusive of loose

surfacing, shall be calculated, "b." The dimensions of two representative shingles shall be accurately measured and the net area of material in each shingle calculated in square feet, "c."

The area in square feet of material contained in each bundle selected shall be calculated ($a \times c$). The net weight of roofing material per 108 sq. ft. contained in each bundle shall be calculated ($\frac{b}{a \times c} \times 108$), and the minimum weight per 108 sq. ft. recorded. This shall be regarded as the minimum weight per 108 sq. ft. of the lot.

The average weight (d) per 108 sq. ft. for the bundles examined shall be calculated. This shall be regarded as the average weight per 108 sq. ft. of the entire shipment. (1a)

The average weight per shingle ($\frac{c \times d}{108}$) shall be calculated (1b)

From the bundles selected, a definite number of representative shingles, whose weight per 108 sq. ft. shall fall within 1.5 per cent of the average weight ascertained in (1a) shall be taken for further examination. The number of shingles so selected shall represent as closely as possible 6 sq. ft. of material, and shall be selected as far as possible from different bundles.

NOTE.—As a referee method, or in case any dispute arises regarding the properties as may be ascertained from the particular sample selected, then a similar sample shall be taken and examined separately from each bundle of shingles selected.

Test 66. Thickness. This determination has been standardized as follows:

(I) *For Felt-base Products:* The thickness of each sample shall be measured at ten equally spaced points at least 1 in. from a cut edge with a dead-weight micrometer gage having a cylindrical foot and anvil with flat bearing surfaces 1 sq. in. in area. The load used shall be 2 lbs. per sq. in. The thickness for each roll sampled shall be averaged and the minimum, maximum, and average thicknesses calculated to the nearest 0.001 in.

(II) *For Paper-base Products:*⁴ These methods cover the procedures for determining the thickness of all kinds of paper and paper products, except crepe paper and corrugated board. Five methods are covered, applicable to the following types of paper:

Method A, for the general run of papers and paper products.

Method B, for the general run of paper products. The use of the spring-actuated instrument is permissible as an alternative to the apparatus prescribed in method A, but is not recommended.

Method C, for soft, low density papers, such as roofing felt.

Method D, for electrical insulating papers.

Method E, for paper 2 mils (0.002 in.) and under in thickness.

Thickness as measured in these methods is defined as the thickness of a single sheet, in contradistinction to "bulking thickness," which is the thickness of a pile of sheets determined in accordance with A.S.T.M. Designation: D 527.

The apparatus shall consist essentially of two plane parallel faces which can be moved apart or together along an axis perpendicular to themselves. In use, one of these faces (the anvil) shall be held stationary, the specimen shall be placed over it, and the other face (the presser foot) which is circular moved towards it until it exerts a predetermined pressure on the specimen. When this condition has been reached, the distance between the two faces shall be read on a suitable device and recorded as the thickness of the specimen.

Method A: The diameter of the presser foot shall be not less than 0.56 in. nor more than 0.65 in. The force shall be exerted by gravity acting on the presser foot and the moving parts connected therewith, and shall be such that the pressure is 8 ± 1 lbs. per sq. in.

Method B: The diameter of the presser foot and the pressure shall be the same as for method A, but the force shall be exerted by a spring instead of by gravity.

Method C: The presser foot and actuating force shall be the same as that prescribed for method A, but the pressure shall be 4 ± 1 lbs. per sq. in.

Method D: A machinists' micrometer conforming to the requirements for the instrument prescribed in method A A.S.T.M. Designation: D 374.

Method E: The apparatus prescribed for either method A or method D may be used.

Place a hard steel ball about $\frac{1}{16}$ in. in diameter fixed firmly in

a thinner flat piece of metal to serve as an anvil and note the readings at different points on the handle.

Set the instrument to zero and place standard gage blocks having an accuracy of 0.00001 in. between the plane faces and observe the corresponding dial readings over the range to be used.

Determine the force required to just prevent the movement of the presser foot from a reading approximately corresponding to the average thickness of the paper to be tested to a lower reading with a suitable balance and determine the contact pressure with this force. For example, attach a fine copper wire to the presser foot where it projects through the top of the apparatus, and, by means of a coarse balance or a calibrated spring, measure the force necessary to prevent the closing of the foot. Alternatively, a stirrup may be used made of a flat metal plate having a hole larger than the diameter of the micrometer foot, covered at the bottom of the plate by a thin metal disk of about the average thickness of the paper to be measured. Suspend the stirrup from a suitable balance.

Calibrate the micrometer used in methods D and E in accordance with the procedure described in A.S.T.M. Designation: D 374.

Ten specimens shall be tested, each having a width of not less than 2 in. If it is not possible to secure specimens 2 in. in width, the width shall be stated in the report. Test specimens shall be taken from samples representative of the shipment and shall be free from creases.

Specimens shall be brought to equilibrium with an atmosphere of known temperature and humidity and shall be tested in that atmosphere.

Methods A, B, and C: Place the specimen between the jaws of the measuring device and lower the presser foot as gently as possible upon the surface of the paper, with its edge at least $\frac{1}{4}$ in. from the edge of the paper. Determine the thickness of each of the ten specimens in each of two different places. If the mean between the maximum and minimum of the 20 results differs from the average of all 20 by more than plus or minus 5 per cent, measure enough additional specimens to obtain agreement within these limits.

Method D: Determine the thickness of electrical insulating papers in accordance with the procedure described in method A of A.S.T.M. Designation: D 374.

Method E: Determine the thickness in accordance with the procedure described. Determine the thickness of at least ten specimens of ten sheets each (Note).

NOTE.—For papers having a thickness of 2 mils or less a single specimen shall consist of a pile of ten sheets. If a micrometer is used to measure the thickness of a single sheet of this thinness, the error in reading the instrument is likely to make a significant difference in the result. If a pile of ten sheets is measured and the result divided by ten, there will also be an error due to the "piling effect," but the latter error will, in general, be smaller than the former.

The report shall include the following:

- (a) Method used for determining thickness,
- (b) Relative humidity and temperature of conditioning atmosphere,
- (c) Number of specimens tested, if more or less than ten,
- (d) Width of the specimens, if less than 2 in., and
- (e) Maximum, minimum, and average thicknesses measured to the nearest 0.0001 in.

Test 67a. Tensile Strength of Bituminized Products.

(1) *For Paper- and Felt-base Products:* With a trimmer having a stop attached exactly 1-in. behind the blade, 10 test specimens shall be cut with the fiber grain, as shown at B-1 to B-10 in Fig. 363, and 10 strips across the fiber grain, as shown at C-1 to C-10 in Fig. 363. Each specimen shall measure 6 in. and have parallel sides 1 in. apart (within $\frac{1}{32}$ in.) with the edges cut straight and clean. Both sets of specimens shall be tested at 70° F. (21.1° C.), using a tension testing machine such as the Scott Strength Tester (Type F, 100-lb. capacity), the Perkins Strip Tester, or equal, in which the clamps are attached to swivels free to move in any direction. The test specimens shall be gripped 1½ in. on each end, leaving approximately 3 in. between the clamps. The tension shall be increased by causing the lower clamp of the machine to travel at a uniform speed of 12 in. per minute. If any specimen breaks nearer than ½ in. to either clamp, the reading shall be disregarded, and an additional specimen shall be tested in its place. The ten readings, with and across the grain, respectively, shall be averaged for each roll sampled. From these results the average strength with and across the fiber grain, respectively, of the fabric as supplied, shall be calculated.

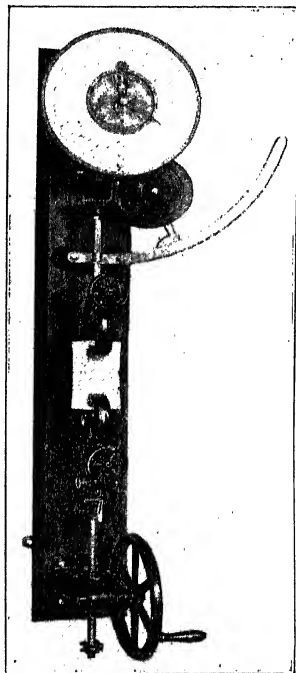
An instrument for ascertaining the tensile strength is illustrated

in Fig. 364, consisting of a pendulum type of machine. The pointer remains in position when the specimen breaks, and is reset by means of the handle suspended from the curved quadrant.

(II) *For Fabric-base Products:* This test shall be conducted as described in Test 79c applicable to the desaturated fabric.

Test 67b. Bursting Strength. The Mullen Tester^a is used for this purpose as illustrated in Fig. 365. This instrument has a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber is filled with glycerin and the test specimen is held in position over the diaphragm in a clamp having a circular hole 1 sq. in. in area, so that the diaphragm will force the material into the hole when pressure is applied under the diaphragm. The pressure is indicated on a dial graduated in 0.5 lb. divisions and during the test is increased at the uniform speed of 2 lb. per second at a uniform temperature of 77° F. Ten tests shall be averaged.

Test 67c. Tearing Strength of Paper- and Felt-base Products. This is determined by means of the Elmendorf Tester illustrated in Fig. 366,^a consisting of a heavy sector *A* carrying a dial graduated from 0 to 100, mounted on ball-bearings on the support *B*. The instrument is leveled by the screw *C* in the base *D*. When the instrument is level, a white mark *E* on the sector will be opposite the end of the spring-stop *H* when the sector is free to swing. To test for zero, push the sector *A* to the left until the spring-stop *H* engages in the right-hand edge of the sector. Set the pointer *K* against the pointer stop *L*, which is fastened to the base by the screw *M*. Release the sector by depressing *H* as far as it will go and hold it down until the sector has swung once in either direction, and then release the

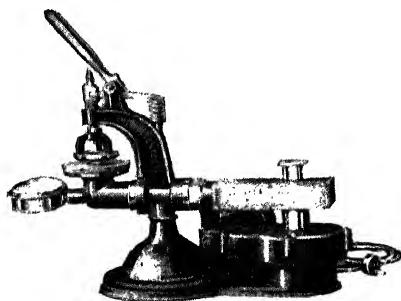


Courtesy Henry L. Scott & Co.

FIG. 364.—Tensile Strength Tester.

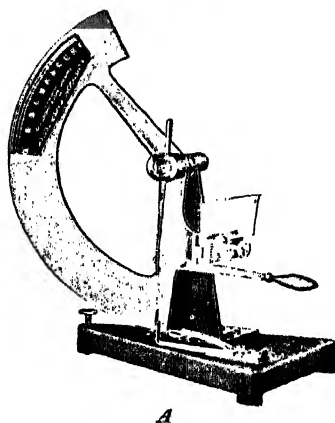
spring-stop. Note the reading of the pointer *K*, and if not at zero, adjust the position of the pointer stop *L* by means of the screw *M*, until the pointer on further trial comes to zero. Insert a sample *P*, 6.3 cm. (2.5 in.) in width and at least 6.3 cm. (2.5 in.) in length, cut with straight edges, in the clamps *N*, and set sector *A* and pointer *K* as in the zero test, then depress the cutter lever *O* to low position and release, thereby cutting the strip a distance of 1.7 in. across the 2.5 in. width. The specimen should be tested at a temperature of 75–

79° F. Depress the spring-stop as far as it will go and hold it down until the sector has swung once in each direction. The distance swung by the sector is indicated by the position of the pointer, which is recorded as a measure of the tearing strength. The ma-

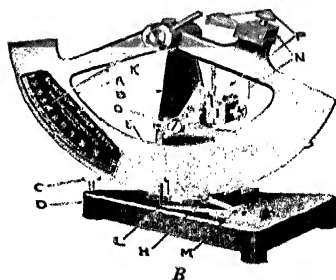


Courtesy B. F. Perkins & Sons, Inc.

FIG. 365.—Bursting Strength Tester.



A



B

Courtesy Thwing Instrument Co.

FIG. 366.—(*A* and *B*).—Tearing Strength Tester.

terial is tested separately with and against the fiber grain. The author has found that in the case of composition roofings, the tear-

ing strength against the fiber grain remains substantially unaffected as the specimen ages, whereas the tearing strength with the fiber grain gradually increases with age, until it eventually exceeds the former.

This test has been standardized as follows:

The testing machine shall be of the pendulum impulse type so designed as to produce a tear approximately 4.2 cm. (1.66 in.) long. The knife mounted on the machine to cut the slit for the tear shall be maintained sharp.

The specimens shall be taken from the original samples and shall be cut 6.3 cm. (2.5 in.) in width and at least 6.3 cm. (2.5 in.) in length. Enough specimens shall be cut so that at least five readings in the machine direction and five readings in the cross-direction, if possible, can be obtained for each original sample.

Enough specimens shall be torn so that the readings on the machine shall be not less than 10 nor more than 20 g., unless one paper alone yields a reading greater than 20 g., in which case only one paper shall be torn at one time. The specimens shall be so arranged that an equal number of tears originating from opposite edges of the specimens will be produced. Readings obtained when a tear deviates more than 6.3 mm. (0.25 in.) from a straight line, shall be rejected. The readings obtained shall be multiplied by the instrument constant corresponding to the number of sheets torn.

The tearing strengths obtained on the machine-direction specimens and on the cross-direction specimens shall be reported separately in grams. The average, the maximum, and the minimum tearing strengths shall be reported for the machine-direction specimens. If possible, similar results shall be reported for the cross-direction specimens.

A test has been standardized for ascertaining the machine direction of paper products.⁷

Test 68. Pliability Test. Two methods have been proposed for this determination, as follows:

(1) *Mandrel Test:* This may be performed as follows:

With the trimmer, five 6-in. strips shall be cut with the fiber grain, as shown at D-1 to D-5 in Fig. 363, each 1-in. in width, and immersed in water at 77° F. (25° C.) for from 10 to 15 minutes. These strips shall be bent through 180 deg. at a uniform speed, in

exactly two seconds, around a mandrel the diameter of which shall be as follows: The first, around a mandrel 25 mm. in diameter; the second, around a mandrel 20 mm. in diameter; the third, around a mandrel 15 mm. in diameter; the fourth, around a mandrel 10 mm. in diameter, and the fifth, around a mandrel 5 mm. in diameter. The test may also be made at 32° F. (0° C.). The pliability shall be expressed numerically from 1 to 10 as follows:

- (1) Cracks entirely through the sheet on the 25-mm. mandrel.
- (2) Cracks part way through the sheet on the 25-mm. mandrel.
- (3) Cracks on the 25-mm. mandrel.
- (4) Cracks on the 20-mm. mandrel.

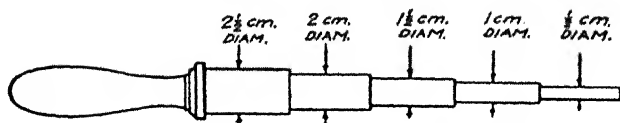


FIG. 367.—Mandrels for Testing Pliability.

- (5) Cracks on the 15-mm. mandrel.
- (6) Cracks on the 10-mm. mandrel.
- (7) Cracks on the 5-mm. mandrel.
- (8) Cracks when bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel.
- (9) May be bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel in one direction without cracking, but will crack when bent through 360 deg. in the opposite direction.
- (10) May be bent through 180 deg. over a 1/16-in. (1.588-mm.) mandrel in one direction and then through 360 deg. over a 1/16-in. (1.588-mm.) mandrel in the opposite direction without cracking.

The readings for each roll shall be averaged separately, and from these results the minimum, maximum and average pliability of the felt shall be calculated.

A convenient instrument for this purpose is illustrated in Fig. 367.

Saturated-felts are usually bent around a 1/16-in. mandrel.

Smooth-roll roofings and mineral-surfaced roll-roofings are tested in accordance with the following standard method:*

Felted Fabrics: From the sample selected, ten test specimens 1 in. in width by 8 in. in length shall be cut from the sample, five in the direction of and five across the fiber grain. They shall be im-

mersed in water at 77° F. (25° C.) for 10 to 15 min., then removed, and each specimen immediately bent 90 deg. over the rounded edge of a block at a uniform speed in approximately 2 sec. The block shall be 3-in. square by 2 in. in thickness with rounded corners of ½-in. radius for 15-lb. felts and smooth-roll roofings, and ¾-in. radius for 30-lb. felts, granular-surfaced roll-roofings and cap-sheets. In bending, the specimen shall be held by hand tightly against the upper 2-in. face of the block, and the projecting end of the specimen shall be bent over the rounded corner without exerting any strain other than that required to keep the specimen in contact with the block and to avoid kinking. Any surface ruptures exceeding ¼ in. in length shall be considered failures.

Woven Fabrics: From the sample selected, five test specimens 1 in. in width by 6 in. in length shall be cut in the direction of the warp. They shall be immersed in a cooling mixture of ice and water at 0° C. (32° F.) for 10 to 15 min., then removed and each specimen immediately bent over a ¼₁₆-in. mandrel through an arc of 180 deg. at a uniform speed in approximately 2 sec. and then through 360 deg. over the same mandrel in the opposite direction. The specimens shall then be dried thoroughly and examined. If one or more of the test specimens crack, ten specimens from another portion of the sample shall be cut and the test repeated. If one or more of these specimens crack, the material shall be considered as failing to conform to the specifications.

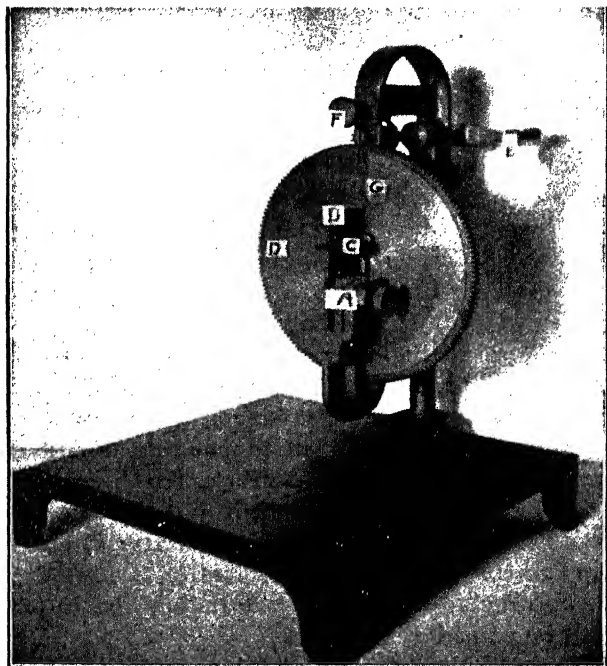
Surface ruptures aggregating more than ¼ in. in length in any direction shall be considered a crack.

(II) *Reeve and Yeager Tester:* This is a more accurate tester, and is illustrated in Fig. 368.⁹

The essential parts of the apparatus are: the clamp, *A*, holding the strip of roofing under test, and the large wheel, *D*, carrying the pin, *C*, 1 inch distant from its center and projecting perpendicularly from its surface. The clamp, *A*, is designed to hold the lower 2 in. of a roofing specimen 4 in. long and 1 in. wide. This clamp is located directly in front of the large wheel so that the back of the clamp, the top of which is rounded to a ¾₁₆-in. radius, serves as a mandrel located on the central line of the bearing on which the large wheel revolves. Thus placed, the pin, *C*, projecting perpen-

dicularly from the face of the large wheel, and the rounded back of the clamp, are parallel.

When a strip of roofing, *B*, is held in the clamp, as shown, rotation of the large wheel brings the pin into contact with that part of



Courtesy A.S.T.M.

FIG. 368.—Apparatus for Measuring the Pliability of Roofing.

the strip projecting above the clamp, and by continuing the rotation, the pin, bearing on the strip, causes it to bend about the $\frac{3}{16}$ -in. rounded back of the clamp. The limit of the rotation is reached when the strip has been bent double over the back of the clamp, practically equivalent to 180 deg. of bend.

The rotation of the large wheel and the consequent bending is readily accomplished and controlled by turning the handle, *E*, operating the small pinion gear in mesh with the large wheel. The rate

of turning is controlled by a metronome to a speed equivalent to bending the specimen through 180 deg. in thirty seconds.

For convenience in measuring the angle through which the strip is bent, the protractor, *G*, graduated in degrees, is fixed to the wheel, *D*, in a suitable position. In starting a test, the pin is brought in contact with the projecting strip of roofing when it is in a vertical position and the pointer shown just below the set screw, *F*, is adjusted to indicate zero. This feature provides for roofings of different thicknesses requiring slightly different initial positions of the large wheel.

As will be seen from Fig. 368, the upper surface of the strip at the point where the bending occurs is at all times clearly visible, so that the development of a break in the coating is readily discerned. When this occurs, the motion of the machine is stopped and the angle of bending read by the indicator and protractor. This value is recorded as a measure of the pliability of the roofing.

To secure reproducible test conditions, the necessary temperature control is obtained by means of a water bath. The large wheel and clamp are carried on a vertical support, so designed that they can be lowered into the water bath, which is placed on the broad base of the machine and which is made high enough to provide a water level just readily inserted and removed without raising apparatus from the control bath.

Test 69. Resistance to Moisture. The following procedures have been standardized:

(1) *Of Bituminized Felts.* Accurately cut a strip of bituminized fabric 18 in. x 18 in., and weigh. Remove the detached mineral particles from both sides of the sheet with a moderately stiff brush, and reweigh (area equals $2\frac{1}{4}$ sq. ft.). Suspend in a tight box containing sufficient water at the bottom to saturate the air with moisture. Cover tightly and allow the specimen to remain in the moist air for 100 hours at 77° F. As the moisture enters more readily through the cut edges of the sheet than through the surface itself, 6 in. should be trimmed from the edges at the termination of the test, leaving a strip measuring exactly 12 in. x 12 in., representing the central portion of the original specimen, and weighing $\frac{2}{3}$ of the latter. Ascertain the weight, thickness and tensile strength of the 12 x 12 portion at the end of the test, and calculate

any variation in percentage from the original figures. The increase in weight should be figured on the basis of the original material *including* the detached mineral matter.

A test has also been proposed for determining the permeability of building papers to water vapor.¹⁰

(II) *Of Textile Products.* A method has been standardized for determining the resistance of the textile fabrics to moisture.¹¹

Test 69a. Water Absorption. A specimen 18-in. square, as shown at *E* in Fig. 363 shall be cut from each sample, weighed, and completely immersed in distilled water at 77° F. (25° C.) for twenty-four hours. The specimen shall then be removed and dried superficially by pressing lightly between two towels. As the moisture enters through the edges of the sheet more rapidly than through the surfaces, each specimen shall be trimmed to exactly 12 in. square, representing four-ninths of the original area, and reweighed rapidly. The increase in weight shall be calculated on the basis of the original test specimen, by multiplying by 2¼. The percentage increase in weight represents the water absorption. The minimum, maximum and average for the shipment shall be calculated.

The results in Table CLII have been obtained upon subjecting representative samples of asphalt-saturated felt, smooth roll-roofing and mineral-surfaced shingles to Tests 69 and 69a, respectively.

Five test specimens shall be cut with the fiber grain as shown at F-1 to F-5 in Fig. 363; five other specimens shall be cut across the fiber grain as shown at G-1 to G-5 in Fig. 363 and the strength shall be redetermined. The decrease in strength in percentage shall be calculated after the specimen has been subjected to water.

Test 70. Blistering Tendency. *Method I:* The following rapid test may be used for ascertaining the blistering tendencies of granular-surfaced bituminized roofings and shingles: The specimen is immersed in water at room temperature for 48 hours, the surface wiped dry with a cloth, and then heated, either: (a) for 2 hours in an air-oven at 175° F.; or else, (b) for 2 minutes' immersion in glycerin at 250° F. The appearance of blisters will be indicative as to the behavior of the roofing on actual exposure.

Method II: The following alternate procedure may be conveniently used either as a study method or as a control method. For

TABLE CLII
RESISTANCE OF ROOFINGS TO MOISTURE AND WATER

Weight in Lbs. per 100 Sq. Ft.	Asphalt- saturated Felt	Smooth Roll-roofing	Mineral- surfaced Shingles
Dry felt.....	11.12	11.12	11.12
Saturant.....	20.88	21.13	21.13
Coatings.....	18.56	16.38
Mineral filler.....	7.99	7.99
Mineral granules.....	23.63
Finished weight.....	32.0	58.8	80.25
Per cent saturation in felt.....	187.5	189.9	189.90
Moisture absorbed (<i>gain % by wt</i>):	(<i>Test 69</i>)		
5 days.....	3.3	0.7	0.6
10 days.....	5.4	1.3	1.1
20 days.....	5.5	2.0	1.9
30 days.....	5.6	2.2	2.1
Water absorbed (<i>gain % by wt</i>):	(<i>Test 69a</i>)		
1 day.....	15.3	2.1	3.2
10 days.....	26.1	9.0	10.2
20 days.....	30.4	11.9	12.0
30 days.....	32.7	13.9	12.6
40 days.....	34.5	15.1	13.1
50 days.....	35.7	16.4	14.3
75 days.....	39.3	19.0	16.3

the latter purpose the test is expedited by exposing the sample to water under vacuum as described below.

This test has not been related to outdoor performance but is intended to determine whether or not roofing of such character is "blisterable."

As a study method it has been found useful in determining the characteristics of the sheet all the way across the machine. When used for this purpose, narrow strips cut across the entire width of the machine may be selected, cut up into small sections which are identified with relation to each other, and after the blister test, put together for observation.

Apparatus: 1 metal pan or dish of sufficient size to accommodate a 4 x 6-in. sample of roofing suspended horizontally with at least a 2-in. total depth of glycerin. (8 in. diameter x 3½ in. deep is satisfactory.)

1 in. laboratory thermometer of suitable range extending to at least 300° F.

1 tripod for above pan.

1 support for thermometer.

1 metal screen carriage for holding 4 x 6-in. sample in place when immersed in glycerin. A convenient carriage was constructed of ½-in. mesh screen using copper wire for a lifting cradle and upper retaining wires to prevent sample from floating off the carriage.

1 stop watch.

Method: The metal pan is filled with glycerin to a depth of at least 2 in. The thermometer is placed in the glycerin which is maintained at $250 \pm 5^{\circ}$ F.

Prior to testing, the roofing may be soaked in tap water for at least 24 hrs. at room temperature or alternatively, for a rapid test, it may be subjected to 15 min. of 28-in. vacuum under water, followed by 5 min. soaking at atmospheric pressure. After removal from the water, the sample is dried superficially on both sides before an electric fan. It is then placed on the screen carriage and immersed horizontally in the hot glycerin bath for 2 min. by the stop watch.

At the end of the prescribed time, the sample is carefully removed from the bath and promptly cooled by plunging carriage and sample into a bath of cold water to preserve the blisters and remove the glycerin. Later the sample is dried at room temperature, exerting every precaution throughout the test to prevent undue loss of loose slate granules.

The degree of blistering may be rated none, trace, slight, severe or excessive.

The following alternate procedure has been proposed: 18 samples measuring 2 by 6 in. are numbered and placed in a constant temperature water bath adjusted to 140° F. (60° C.). After ¼ hr. 3 specimens are removed from the water bath, dried superficially with a paper towel and placed before a fan until the surfacing is dry. They are then placed in a closed container with water in the bottom to maintain an atmosphere of 100 per cent relative humidity and so as not to contact the water in the bottom. This prevents drying out or losing water when the specimens are waiting to be placed in the oven. After ½ hr. a second group of 3 specimens is removed from the water and the above procedure

repeated at 15-min. intervals. The 18 specimens are then removed from the closed container and placed in an electric oven measuring 12 by 12 by 12 in., provided with a circulating fan at the top and with extra heating elements in addition to the regular thermostatically controlled elements, which can be turned on temporarily. The electric oven is maintained at 220° F. (104.4° C.) for one hour. The 18 specimens are placed on 3 shelves provided in the top part of the oven, set at an angle of 45 to 60 deg. and with the aid of the circulating fan heated uniformly. Due to the cooling effect of the specimens, the additional heating elements are turned on when the specimens are placed in the oven, so that the temperature should return to 220° F. in 8 to 10 min. The first 15 min. heating, therefore, ranges from 190 to 220° F. The blistered condition of the specimens is graded according to the following classification:

No blisters	None
1 to 5 blisters per 12 sq. in.	Trace
6 to 10 blisters per 12 sq. in.	Slight
11 to 25 blisters per 12 sq. in.	Moderate
26 to 50 blisters per 12 sq. in.	Severe
More than 50 blisters per 12 sq. in.	Excessive

Test 71. Loss on Heating. The following methods have been proposed:

(I) *For Asphalt-saturated Fabrics:* From each sample, 12 by 6-in. specimens shall be cut at K-1 and K-2 in Fig. 363, care being taken not to disturb any of the detached surfacing. Each specimen shall be weighed and suspended in the center of an air-oven maintained at 221° F. (105° C.) \pm 5° F. (3° C.) by means of a thin wire fastened through holes punctured near one edge. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The specimens shall be kept in the oven for exactly five hours, then cooled and removed carefully, and each specimen weighed. The average loss shall be calculated as a percentage and the average percentage of moisture deducted. The final figure shall represent the average loss on heating, exclusive of moisture.

(II) *For Coal-tar Saturated Felt Only:* From the rolls selected, strips 6 in. wide shall be cut across the sheet. Sufficient of these shall be taken to make up a sample of 250 g. \pm 5.0 g. These strips then shall be rolled up and placed in the wire basket of the

extraction apparatus described in Test 59. The sample shall then be covered with a disk of soft filter paper to insure an even distribution of the solvent.

The carbon disulfide extract containing the bitumen shall be transferred to a 500 ml., short-neck, round-bottom flask. The flask shall be equipped with a Hempel column, 16 cm. in length, 15 mm. in inside diameter and filled to a depth of 5 cm. with glass beads. The Hempel column shall be connected with a water-cooled condenser. A -2 to $+80^{\circ}$ C. thermometer shall be placed in the Hempel column so that the top of the bulb shall be opposite the bottom of the tubulature. Distillation for the removal of carbon disulfide shall be continued by means of a steam bath until drops cease to fall from the condenser. The distillation then shall be carried out over a small flame, heating carefully and observing the thermometer until the distillation ceases and the temperature of the vapor as indicated by the thermometer shows no further rise on slightly increasing the flame.

When the contents of the flask are sufficiently cool to be poured without appreciable vaporizing, 100 g. \pm 0.1 g. shall be distilled in accordance with Test 16b.

Fractions shall be taken at 210° C. and 235° C. and the distillation stopped at that point. The per cent of distillate by weight at each temperature shall be calculated on the basis of the weight of sample taken for distillation.

(III) *For Asphalt Roll-roofings and Shingles:* Two strips shall be cut from the sample of roofing or shingles, each approximately 8 by 8 in. The specimens shall be weighed and then suspended vertically, in the same direction as the material would be applied to the roof, in the center of an air-oven maintained at 176° F. (80° C.) $\pm 5^{\circ}$ F. (3° C.) with a thin wire fastened through holes punctured near one edge. The internal dimensions of the oven shall not be less than 12 by 12 by 12 in. An electrically heated oven is recommended. The thermometer shall be inserted in the oven to such a depth that its bulb will be in line with the center of the specimens. The specimens shall be maintained at the prescribed temperature for exactly two hours, then cooled in a desiccator, and each specimen weighed. The average loss of volatile matter shall be calculated as a percentage. Any change in appearance of the speci-

men shall be recorded, such as flowing, sagging, blistering or absorption of the asphalt coatings; also sliding of granular surfacing.

An instrument has been described for testing the heat-resistance of roofings by recording the rise in temperature upon subjecting the surface to radiant heat.¹²

Test 71a. Discoloration Tendency. An "exudation test" to ascertain whether or not asphalt roofings will discolor on aging (due to the incompatibility of the asphalts used) has been described as follows:¹³

The coating is warmed to a fluid condition. It may then be poured into the lid of a 3-oz. (88.7-ml.) penetration tin or other convenient receptacle in a layer 0.3 to 0.6 cm. (0.125 to 0.25 in.) thick. To remove air bubbles the surface of the coating may be momentarily heated. The surface area and total weight of the specimen are determined and the surface is then given a preliminary dusting with fine roofer's talc, evenly distributed over the surface, neither the surface nor the talc being handled by the fingers during this operation. The excess of nonadherent powder is removed by inverting the specimen and allowing the container to drop 2.5 cm. (1 in.) onto the table top. A second application of fine talc is then made by gently shaking or tapping a 300-mesh sieve held 7.5 cm. (3 in.) above the surface of the specimen, so that a fine mist rather than agglomerated particles of the powder may accumulate on the specimen. This operation is continued with occasional weighings until a uniform film of talc weighing 0.025 g. per sq. in. (6.45 sq. cm.) has been obtained. Uniformity in the thickness of the talc film is of great importance in obtaining reproducible results, for the thicker the layer of talc (up to a certain limit), the wider will be the ring formed.

A drop of the saturant about 0.16 cm. (0.0625 in.) in diameter is placed upon the talc-dusted surface of the coating. This may be done most conveniently by plunging the end of a heated spatula or paring knife into the cold saturant and, after the excess has drained off, allowing a drop of suitable size to fall on the dusted surface from a height of about 1.25 cm. (0.5 in.). Several drops of the same or different saturants may be applied to a single specimen of dusted coating.

The specimen is then placed in an oven maintained at a tem-

perature of $43.33 \pm 2.8^{\circ} \text{C.}$ ($110 \pm 5^{\circ} \text{F.}$), for a period of 72 hours. With some asphalts that are entirely free from strike-through tendencies towards each other, no reaction whatever will occur in this test, except for the very slow flattening of the spherical drop and the gradual yellowing of the dusting. With other asphalts that do have strike-through tendencies, the drop will flatten more rapidly, and relatively early in the test a thin ring of a darker color than the surrounding area will form on the dusted surface right around the periphery of the drop, and will grow wider, blacker, and glossier, till it reaches a maximum width and gloss characteristic of that combination of asphalts, and of that type and quantity of dusting, after which it spreads and darkens no further.

The average width of the ring of discolored talc around the periphery of the spot is measured to the closest 0.1 mm. by means of a magnifying-glass, and furnishes an "index of bleeding."

Test 72. Electrical Tests. The following tests have been standardized, a full description of which will be found upon referring to the original sources:

Dielectric Strength.

Of sheet insulating materials.¹⁴

Of laminated sheet insulating materials.¹⁵

Of adhesive insulating tape.¹⁶

Of varnished cloths and varnished cloth tapes.¹⁷

Test 73. Special Tests Applicable to Insulating Tape. The following tests have been standardized for adhesive insulating tape:¹⁸

These specifications cover friction tape commonly used for protecting and binding in place, insulation applied to joints of electrical wires and cables, and for other electrical and mechanical purposes. The tape consists of cotton sheeting that has been impregnated with an adhesive insulating compound and cut into rolls of narrow width.

The cotton sheeting shall be evenly and firmly woven from good cotton and as free from unsightly defects, dirt, knots, lumps, and irregularities of twist, as is consistent with the best manufacturing practice. The threads shall run in as straight lines as possible without waving, so as to reduce to a minimum the raveling of the cloth when cut into tape.

The frictioning compound shall be a tacky adhesive insulating compound containing practically no free sulfur or other substances which would have a deteriorating effect on copper or other metals, or on the fabric.

The fabric shall be thoroughly impregnated and evenly covered on both sides with the frictioning compound.

The compound shall adhere firmly to the fabric, and shall not pull away from the fabric so as to leave bare spots when adjacent layers of tape are separated.

The tape shall show tackiness, that is, ability to stick to itself after light contact has been made, in the following combinations: (1) front to front, (2) back to back, and (3) back to front when tested in accordance with the procedure described.

The number of pinholes in a specimen of three consecutive yards of tape selected at random from any portion of the sample roll shall not exceed the following:

Tape Width, in.	Number of Pinholes, max.
$\frac{3}{4}$	6
1.....	8
$1\frac{1}{2}$	12
2.....	16

The tensile strength of the tape specimen shall be not less than 40 lbs. per inch of width. The initial distance between the jaws of the testing machine shall be 12 in. and the rate of separation of the jaws shall be 20 in. per min.

Each roll selected for purpose of tests shall be tested for dielectric strength. The breakdown voltage shall be not less than 1000 volts.

Each roll selected for purpose of tests shall be tested for parallelism of the warp threads with the longitudinal axis in accordance with the procedure described. The difference between the compared widths shall not exceed $\frac{1}{8}$ in.

The tape shall have a nominal thickness of 0.015 in. and shall be made in the following widths: $\frac{3}{4}$ in., 1 in., $1\frac{1}{2}$ in., and 2 in. Each roll shall contain not less than 82½ ft. and not more than 85 ft. of tape.

The thickness shall not vary from that specified by more than plus or minus 0.003 in., nor the width by more than plus or minus $\frac{1}{32}$ in.

Each roll shall be wrapped in paraffin paper or its equivalent, or metal foil and, unless otherwise specified, enclosed in a suitable box. The wrapping shall be secure and shall thoroughly protect the contents.

Each box shall be marked with the name of the manufacturer or trade-mark, together with the nominal width and length of the tape.

The tape shall be inspected and tested either at the place of manufacture prior to shipment or at the place of delivery within 4 weeks from date of delivery.

The tape shall be stored in the original boxes and preferably in a cool, dark location. Tape shall not be stored in close proximity to steam pipes, radiators, or other sources of heat.

The tape offered for inspection shall be divided into one or more lots of approximately equal numbers of rolls in accordance with the following requirements:

Rolls Offered for Inspection	Number of Lots
Up to 250.....	1
251 to 750.....	2
751 to 1500.....	3
1501 to 3000.....	4
3001 to 5000.....	5
Over 5000.....	5 plus 1 for each additional 1000 rolls.

The lots shall be marked for identification and one sample roll shall be taken at random from each lot for purpose of tests and marked to correspond with the lot from which it was selected. Unless otherwise specified, one specimen from each sample roll shall be tested in accordance with the specifications.

At least 2 ft. of the outer layer of each roll selected for purpose of tests shall be removed and discarded before taking test specimens.

The tape required for test specimens shall be unwound from the roll at a slow uniform rate without jerking.

(a) Cold Adhesion: The adhesion between adjacent layers of the tape shall be determined as follows: A specimen 30 in. in length

shall be removed from the sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One end of the specimen shall be inserted in the slot of the mandrel described below and 2 in. of the tape shall be wound on the mandrel. A weight of 10 lbs. per inch of width shall then be attached to the end of the specimen and 25 in. of the tape shall be wound on the mandrel at a uniform rate of 12 in. per min. The tape shall be allowed to remain 3 min. with the weight attached, after which a weight of 4 lbs. per inch of width of tape shall be substituted for

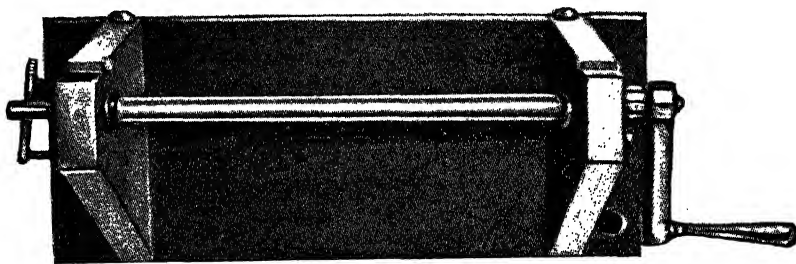


FIG. 369. Tape Tester.

Courtesy A.S.T.M.

the weight of 10 lbs. per inch of width, and the tape allowed to unwind. After the first 2 in. have unwound, the length that unwinds in the first 1-min. interval shall be measured and recorded. This length shall not exceed 15 in. The temperature of the room and the temperature of the tape shall be not less than 20° C. (68° F.) nor more than 22.2° C. (72° F.), except that tape meeting the adhesion test when tested at a higher room temperature shall not be rejected. The relative humidity at the temperature tested shall not exceed 80 per cent.

The mandrel used shall be $\frac{1}{4}$ in. in diameter with a slot approximately $\frac{1}{16}$ in. in width and long enough to accommodate the full width of tape, and shall be mounted in a level position in ball bearings of the Fafnir Bearing Co., Catalog No. 36, Serial No. 30, extra small, or equivalent ball bearings. The mandrel shall turn freely under a weight of $\frac{1}{4}$ oz., suspended from a cotton thread wound in a single layer on the center of the mandrel. Fig. 369 shows a suggested form of tape tester.

Two 8-in. specimens shall be cut from each sample roll, care being taken not to touch the surface to be tested with the hands or otherwise. One specimen shall be placed lightly on a clean horizontal surface, then the second placed evenly upon the first. No weight or pressure other than the weight of the specimen shall be applied to the tapes. The two specimens shall be picked up from one end and then stripped slowly from each other starting at the same end.

The tackiness specified shall be such that the point of separation remains approximately in the same horizontal plane as the hands that pull the tape apart.

The tackiness shall be determined three times and if more than one test fails to conform to the requirements specified, the tape shall be considered as not conforming to the tackiness requirement.

New, clean specimens shall be taken for all tackiness tests. The temperature of the room and the temperature of the tape shall be not less than 20° C. (68° F.) nor more than 22.2° C. (72° F.), except that tape meeting the tackiness test when tested at a higher room temperature shall not be rejected. The relative humidity at the test temperature shall not exceed 80 per cent.

(b) The number of pinholes shall be determined while the tape specimen is held over a slot in the top of an illuminated box. The box shall be approximately 8 in. in width by 8 in. in height and 18 in. in length, inside dimensions. The slot shall be 2 in. in width by 12 in. in length. The box shall be painted white inside and illuminated by a 25-watt lamp. The slot shall be covered with clear glass set flush with the top of the box. Means shall be provided to limit the light to the width of the tape. Ruptures of the insulating film at the extreme edges of the tape, due to the slight tearing action of the cutting knife, shall not be considered pinholes.

(c) Dielectric strength shall be determined by placing a specimen approximately 6 in. in length between two flat electrodes $\frac{1}{4}$ in. in width by $4\frac{1}{4}$ in. in length. The edges shall be square and the ends rounded to $\frac{1}{8}$ -in. radius cylinders whose axes are parallel to the contact faces of the electrode thus giving an area of contact with the surfaces of the tape of exactly $\frac{1}{4}$ in. in width by 4 in. in length. Under an electrode pressure of 1 lb. per sq. in., a 60-cycle alternating-current voltage of practically sine wave form shall be

applied at a value not exceeding 100 v. and raised at a rate of 100 v. per sec. until puncture occurs. In order to prevent flashover, increased width may be secured by attaching to each side of the test specimen an added piece of tape, making a $\frac{1}{8}$ -in. lap seam carefully rolled down.

(d) The parallelism test of the tape shall be made as follows: A piece of tape 16 in. in length shall be cut from the sample roll and then torn lengthwise, assisting the tearing by first slitting one end for about $\frac{1}{2}$ in. with a knife. A 2-in. length shall then be cut from each end of one of the torn halves and the ends of the remaining 12-in. length folded together and compared.

(e) The thickness test shall be determined by means of a micrometer graduated to 0.001 in. and having a presser foot 0.25 ± 0.01 in. in diameter, exerting a total force of 9 ± 0.1 oz. The load shall be applied by means of a weight. Five thickness and five width measurements shall be made at random in a length of not less than 3 ft. on each sample roll and the maximum, minimum, and average of these five measurements shall be reported.

(B) SEPARATION OF FINISHED PRODUCT INTO ITS COMPONENT PARTS

SEPARATION OF BITUMINOUS MATTER, MINERAL MATTER AND FIBROUS CONSTITUENTS

Test 74. Moisture. From each sample, 2-in. test specimens shall be cut, as shown at A-1 and A-2 in Fig. 363. The 2-in. specimens shall be cut into 1-in. squares. About 50 g., selected at random, shall be accurately weighed and distilled with 100 ml. of a coal-tar distillate of which 5 to 10 per cent boils below 100° C. (212° F.) and of which at least 90 per cent distills up to 180° C. (356° F.), in an apparatus of the form and under the conditions prescribed in Test 25b. The flask containing the solvent and sample shall be heated in a paraffin bath at a temperature of 302 to 338° F. (150 to 170° C.) until no further water passes over. The distillate shall be allowed to stand in the graduated receiver until the water separates, whereupon the volume of water shall be measured and its weight calculated. From this, the average percentage of moisture in the fabric, as received, shall be calculated.

Test 75. Analysis of Saturated Fabrics (Single-layered). The following method has been proposed:¹⁸

From each roll selected, a 2-in. strip (within $\frac{1}{32}$ in.) shall be cut across the specimen as shown at H in Fig. 363.

Each strip shall be extracted with carbon disulfide in an extractor shown in Fig. 353, the extraction being continued for several hours after the drippings have become colorless. The desaturated fabric shall be removed and heated in a ventilated oven at 225° F. (107.1° C.), cooled in a desiccator, any adhering comminuted surfacing being brushed off and retained, and weighed as rapidly as possible. The heating shall be repeated until the weight of the fabric remains constant, as determined by two consecutive weighings taken not less than ten minutes apart, which shall show a further loss of not more than 0.1 per cent. This will give the weight of the fabric in the moisture-free state. Where a coal-tar pitch saturant has been used, the moisture-free weight of the desaturated fabric shall be corrected for the carbonaceous matter retained mechanically in its interstices, by means of the method described in the following paragraph.

The following colorimetric method shall be used to ascertain the carbonaceous matter derived from a coal-tar pitch saturant and retained by the desaturated fabric:

(a) About 15 g. of an unsaturated fabric of the same general character as the one under examination, shall be macerated by boiling in water, disintegrating with a rotary egg-beater and picking the fibers apart with needles. The fibers shall be filtered through fine cloth and dried to constant weight at 225° F. (107.1° C.). A 1-g. portion of the fibers shall be accurately weighed into a flask and diluted to exactly 100 ml. with distilled water at room temperature. About 50 g. of glass beads shall be added and the contents of the flask shaken vigorously until the fibers are reduced to a homogeneous pulp in uniform suspension.

(b) A distilled coal tar, having approximately 10 to 25 per cent of insoluble carbonaceous matter, shall be procured and the carbonaceous matter extracted with benzol until it is free from soluble matter; then dried to constant weight at 225° F. (107.1° C.). A 1-g. portion of the purified carbonaceous matter shall be accurately weighed and diluted to exactly 100 ml. at room temperature with a starch solution of a consistency sufficient to carry the

carbonaceous matter into temporary suspension. (A 12.5 per cent solution is recommended.)

(c) The liquid carrying the fibers, obtained as described in Paragraph (a), shall be titrated with the suspension of carbonaceous matter, obtained as described in Paragraph (b), and from time to time a field prepared from a drop of the well agitated mixture shall be examined under a microscope at 100 diameters magnification until the color exactly matches a field prepared from the desaturated fabric under examination, when both are reviewed side by side under parallel conditions. The end-point is fairly sharply defined. The burette reading gives directly the percentage of carbonaceous matter adhering to the moisture-free fabric under examination.

(d) The weight of moisture-free fabric shall be corrected by deducting the weight of adhering carbonaceous matter.

The total comminuted surfacing plus any filler present in the bituminous saturant represents the sum of the detached comminuted surfacing, the amount brushed off from the surface of the desaturated fabric, plus the amount recovered upon evaporating and igniting the bituminous extract obtained as above.

The weight of bituminous saturant represents the difference between the weight of the original fabric, and the sum of the weights of the moisture-free desaturated fabric, plus moisture, plus the total comminuted surfacing and filler present.

An approximate method of determining the impregnation of tarred felt from the known weight of the dry felt (moisture-free), and its ash content, consists in finding the ash from a measured and weighed strip of the saturated felt, from which the weight of impregnation is calculated by difference.¹⁹

Ash: A representative sample shall be secured by cutting from each strip of desaturated felt a piece about $\frac{1}{2}$ in. in diameter as shown at I-1, I-2, and I-3 (Fig. 363). About 25 g. selected at random from all the specimens sampled in this manner shall be accurately weighed, and incinerated in a weighed porcelain or quartz crucible either over an open flame or in a muffle, until all the carbon is consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, and weighed. The percentage of ash shall be calculated on the basis of the moisture-free felt.

NOTE.—As a referee method, or in case greater accuracy is desired, the three portions taken from each specimen roll shall be weighed and ignited separately. The minimum, maximum and average of ash present shall be calculated on the basis of the moisture-free felt.

Test 76. Analysis of Saturated and Coated Fabrics (Single-layered). Since the fabrics and bituminous matter may be assem-

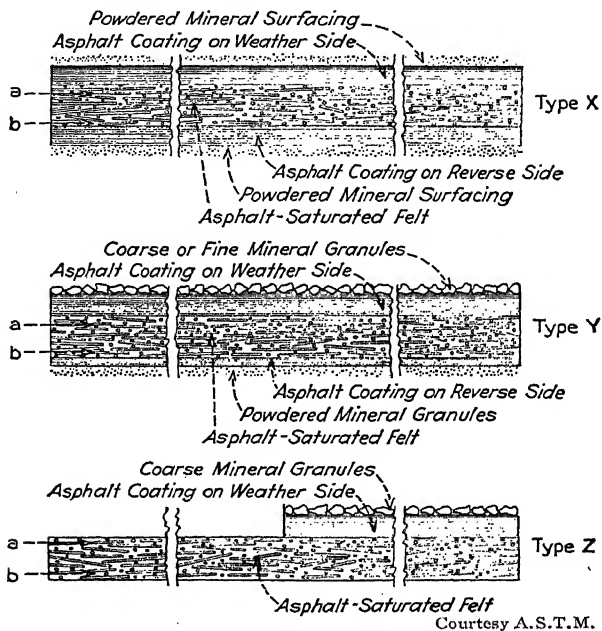


FIG. 370.—Construction of Asphalt Roll-roofing, Cap Sheets, and Shingles.

bled in many different ways, the resultant products are too numerous to itemize. It is impractical, therefore, to give analytical methods applicable to each. The ones which follow have been devised specifically for examining prepared roofings and composition shingles composed of roofing-felt, saturated and coated on both sides with asphalt, and surfaced on the top with either powdered or granular mineral matter, and on the under side with powdered mineral matter. These methods are typical ones, and with slight modifications and a little ingenuity may be adapted to other forms of bituminized fabrics. They have been standardized as follows: ²⁰

These methods cover the procedures for the physical testing and chemical examination of roofing and shingles composed of asphalt-saturated roofing felt coated to various extents with asphalt and the coated portion surfaced with mineral powders or granules.

Asphalt roll roofings, cap sheets, and shingles may be divided into three types (see Fig. 370), as follows:

Type X.—A single thickness of asphalt-saturated felt, coated with asphalt compounded with a finely powdered mineral filler and surfaced with powdered mineral matter such as talc or mica.

Type Y.—Similar to type X, but with the asphalt coating on the weather side surfaced with coarse granules.

Type Z.—A single thickness of asphalt-saturated roofing felt, coated on the weather side for approximately one-half of its width with asphalt compounded with fine mineral matter, and the coated portion surfaced with coarse mineral granules.

The mineral matter, bituminous matter and fibrous matter may be distributed in the following manner:

A—MINERAL MATTER

1. Detached. *Fine Mineral Matter* (e.g., finely ground talc or mica), Type X (on top and bottom), also Types Y and Z (on bottom only).
2. Embedded in the top coating. } *Moderately Coarse Mineral Matter* (e.g., coarsely ground talc) Type X (on top only).
3. Embedded in the bottom coating *Coarse Mineral Matter* (e.g., crushed slate, crushed brick or tile), Types Y and Z (on top only).
4. Admixed with the top coating (Types X, Y and Z) May or may not be present. If present, consists of very fine mineral matter (e.g., clay, silica, limestone, slate flour, shale, etc.).
5. Admixed with the bottom coating (Types X and Y)
6. Ash present in desaturated felt (i.e., ash on incineration).

B—BITUMINOUS MATTER

1. Contained in the top coating.
2. Contained in the bottom coating.
3. Saturant of the felt.

C—FIBROUS MATTER

1. The desaturated felt.

The test strips for the separation of prepared roofing and shingles into their component parts shall be prepared as follows:

The composition of the roofing shall be analyzed as follows:

- (a) Weight of dry felt per 108 sq. ft.,
- (b) Weight of saturant (soluble in carbon disulfide) per 108 sq. ft.,

- (c) Weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),
- (d) Weight of reverse side coating (soluble in carbon disulfide) per 108 sq. ft. (which may be either side on Type X),
- (e) Weight of mineral matter per 108 sq. ft. passing a No. 6 (3360-micron) sieve and retained on a No. 100 (149-micron) sieve, and
- (f) Weight of mineral matter per 108 sq. ft. passing a No. 100 (149-micron) sieve.

From the samples representing the average weight, a number of strips shall be cut, each measuring approximately 2 in. in width by 8 in. in length. These shall be weighed, the length and width measured to within 1 mm. or $\frac{1}{32}$ in., and the weight calculated per 108 sq. ft. Any of the 2 by 8-in. strips whose weight varies more than 1.5 per cent from the average weight of the shipment, shall be rejected. This process shall be continued until eight acceptable specimens are obtained, which shall be used for determining the composition of the roofing.

NOTE.—If a strip is cut to exactly 2 by 7.46 in. its weight in grams divided by two gives directly the weight in pounds per 108 sq. ft.

Detached Mineral Matter (A-1): Remove the detached mineral particles from both sides of the 2-in. strips with a jet of compressed air operating under a nozzle pressure of 25 lbs. per sq. in. and reweigh in grams. Make a correction to include any loose mineral matter (see Test 65). Calculate the weight in pounds per 108 sq. ft. of the detached mineral matter (A-1) (2)

Another means to remove the detached mineral matter²¹ involves the use of a machine as illustrated in Fig. 371. This machine provides an abrasive or scrubbing action on the embedded granules by means of a standardized steel-wire brush actuated by a horizontally reciprocating crosshead driven by a motor with reduction gears. The brush is mounted on a floating head attached to the crosshead by ball-bearing toggles. The length of stroke of the brush is $6\frac{3}{4}$ in. and the load on the brush is approximately 4 lb. per sq. in. of its over-all area (1.25 sq. in.). The recommended test is 50 cycles (each of one forward and one backward stroke), weighing the specimens before and after the test to determine the granule loss. The machine is equipped with a starting device and counter which automatically stops the brush after 50 cycles.

Figure 371 illustrates the assembled machine, showing the floating head and brush lifted out of contact with the roofing test specimen. During an abrasion test the brush is dropped into contact

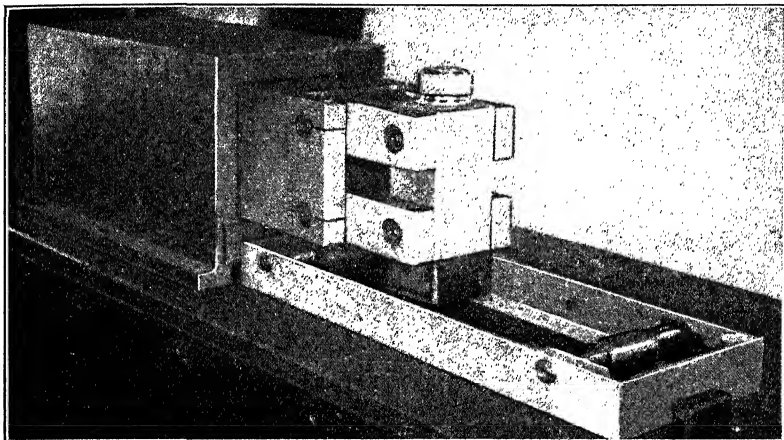


FIG. 371.—Rub-test Machine, Type I.

Courtesy A.S.T.M.

with the specimen. The specimen holder is a cast aluminum-alloy pan, with built-in cam clamps, so that the specimen may, if desired, be kept immersed in water during the abrasion test. In making the "wet" test, it is preferable to collect, dry and weigh the granules

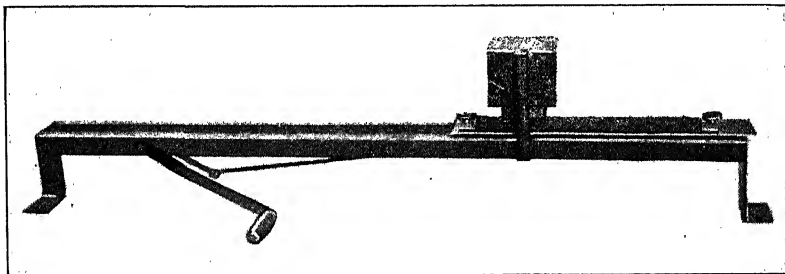


FIG. 372.—Rub-test Machine, Type II.

Courtesy A.S.T.M.

removed, rather than to attempt to dry and weigh the specimen. A simpler type of machine is illustrated in Fig. 372. This machine also employs a steel-wire brush, the length of stroke and pressure

being approximately the same as in the preceding, but is operated by hand instead of by a motor, and requires counting of the number of strokes, since it has no automatic stopping device. It also differs in that the specimen moves instead of the brush. The test may either be run "dry," as shown in the illustration, or by using a pan, may be run "wet," with the specimen immersed in water during the test.

Moisture-free Felt (C-1): From the sample representing the average weight of the lot of roofing, specimens shall be cut, each measuring approximately 2 in. in width by 8 in. in length. These specimens shall be weighed, the length and width measured to within $\frac{1}{32}$ in., and the weight calculated per 108 sq. ft. Any of the 2 by 8-in. specimens whose weight varies more than 1.5 per cent from the average weight of the lot is extracted with carbon disulfide in a suitable extractor or centrifuge until the washings are colorless. (Benzol having a boiling point of 80° to 82° C., carbon tetrachloride, or chloroform may be used if desired, but in case of dispute carbon disulfide shall be used.) The desaturated felt shall be dried in air, then placed in a tared weighing bottle and further dried at 105° to 110° C. (221° to 230° F.) for 30 min., and weighed. The heating shall be repeated until the weight remains constant, as determined by two consecutive weighings taken not less than ten minutes apart, which shall show a further loss of not more than 0.1 per cent. Any adhering mineral matter shall be brushed off the felt, weighed and retained for further examination. The weight of said adhering mineral matter shall be deducted to obtain the weight of the moisture-free felt. The extract and accompanying mineral matter shall be retained for further examination. From the weights so obtained and the respective areas of the strips of desaturated felt, the weight of moisture-free felt shall be computed in pounds per 108 sq. ft., from the following formulas and the results averaged:..... (3)

$$\left. \begin{array}{l} \text{Pounds per} \\ 108 \text{ sq. ft.} \end{array} \right\} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of the strips in square centimeters}} \times 221.2$$

or

$$\left. \begin{array}{l} \text{Pounds per} \\ 108 \text{ sq. ft.} \end{array} \right\} = \frac{\text{Weight of moisture-free felt in grams}}{\text{Area of the strips in square inches}} \times 34.3$$

The extracted felt shall be retained for examination as to weight, thickness, strength, etc., in accordance with the tests described under Section (E).

Ash (A-6) : The following methods have been proposed :

Method (I) : The desaturated felt obtained from the center, the weather and the reverse sides shall be ashed separately in weighed crucibles, either over an open flame or in a muffle furnace, until all carbon has been consumed. A few drops of ammonium carbonate solution shall be added, the sample ignited gently, cooled and reweighed. The percentage of ash in the center portion will be assumed to be the true percentage of ash of the entire felt. The excess ash in the weather side portion will be added to the mineral matter passing a No. 100 (149-micron) sieve as determined. This weight will also be used for correcting the total weight of desaturated felt and weight of saturant soluble in carbon disulfide. In like manner, the excess ash in the reverse side portion will be added to the mineral matter passing a No. 100 sieve and used for correcting the total weights of felt and saturant soluble in carbon disulfide.

The percentage of ash shall be calculated on the basis of the moisture-free felt. (4)

Method (II) : The following has been standardized for determining the ash content of paper and paper products : ²²

This method of test is intended for use in determining the ash content of paper and paper products, which is defined as the residue after complete combustion of the paper. The mineral content of paper may consist of: (1) various residues from chemicals used in its manufacture, (2) metallic matter from piping and machinery, and (3) filling, coating, and pigmenting materials. Generally, if the ash content does not exceed about 2 per cent, no filling, coating, or pigmenting material has been used; but this is not always the case, as pigments of high opacity or coloring power are sometimes used in very small amounts. When filling and coating materials are present that do not change much on ignition, such as barium sulfate and certain titanium pigments, the ash is an approximate measure of the amount present.

The apparatus shall consist of the following:

(a) Crucible: A crucible, made of material such as platinum, alundum, porcelain, or silica, that does not change in weight under the ignition conditions, and having a tightly fitting lid.

(b) Heat Source: An electric muffle furnace with an operating temperature of approximately 925°C . (1700°F .) is recommended, but a gas burner yielding a similar temperature is also suitable.

The test specimen shall consist of small pieces of paper so selected as to be representative of the sample. Its total weight shall be not less than 1 g.

Dry the specimen to constant weight at $105 \pm 2^{\circ}\text{C}$. and weigh to the nearest 1 mg. This may be done with sufficient accuracy for the purpose in the ignited and weighed crucible used for the ashing of the specimen.

Ignite the dried specimen in the crucible, which, together with the cover, has previously been ignited and weighed. To avoid loss of small particles, the crucible shall be covered during the initial ignition of the specimen, which shall be done at low temperature. The temperature shall then be gradually raised to a maximum of approximately 925°C . (1700°F .). After this temperature is reached, the lid of the crucible may be slid to one side until the combustion is complete. Care shall be taken at all times to protect the contents of the crucible from air drafts. When the specimen is completely burned, as indicated by absence of black particles, remove the covered crucible to a desiccator, and allow it to remain until its temperature is in equilibrium with that of the surrounding atmosphere. Weigh the crucible and contents to the nearest 0.1 mg. Repeat the ignition and weighing until the weight is constant.

The percentage of ash shall be calculated on the basis of the weight of the specimen dried to constant weight at $105 \pm 2^{\circ}\text{C}$.

The percentage of ash shall be reported as the average of at least two determinations. Ash shall be reported to the nearest 0.05 per cent for papers containing 5 per cent ash or less, to the nearest 0.1 per cent for papers containing 5 to 10 per cent ash, and to the nearest 0.2 per cent for papers containing more than 10 per cent ash.

Results of duplicate determinations should agree within the following:

Ash Content	Permissible Difference
5 per cent or less	0.1
Over 5 to 10 per cent.	0.15
Over 10 per cent.	0.2

Bituminous Saturation (B-3) in Moisture-free Felt: The following methods have been proposed:

Method (I): Two of the weighed 2 by 8-in. specimens shall each be separated into three horizontal sections approximately at the points indicated by the arrows *a* and *b* in Fig. 370. (These strips may be cut to smaller size and into disks if so desired.) The asphalt coatings with attached mineral surfacings shall be removed in such a manner that some of the saturated felt adheres to them leaving a central section of saturated felt free from coating. Any mineral surfacing detached during this operation shall be added to the section to which it belongs.

The top bituminous coating, with the embedded mineral matter, shall be removed first.

NOTE.—The purpose shall be to remove the entire coatings with some of the saturated felt adhering and no coatings left on the central section of saturated felt. The operator may accomplish this in any way that is most convenient. Care shall be taken to preserve the mineral surfacing with the layers to which it belongs if it becomes detached during the operation. A convenient manner to effect the separation consists in warming the strips in an air-oven at a temperature of approximately 176° F. (80° C.); then with a knife, the front and back coatings, respectively, shall be peeled off, care being taken to remove as little as possible of the saturated felt, and at the same time to make sure that all of the coatings and surfacings are stripped from the felt.

These three horizontal sections thus obtained shall be weighed separately and the total weight should agree within 1 per cent with the original weight of the strips which have been separated. The saturated felt (middle section) shall be extracted with benzol (boiling-point 80 to 82° C.) or C.P. carbon disulfide, in a suitable extractor or centrifuge and the extraction continued until the washings have become colorless. The desaturated felt shall be removed from the extractor, placed in a tared weighing bottle provided with a ground-glass stopper, heated in a ventilated oven at $225 \pm 5^\circ$ F. ($107 \pm 3^\circ$ C.) for 30 minutes, the stopper inserted, then cooled and weighed. The heating shall be repeated until the weight of the felt remains constant as determined by two consecutive weighings taken with an intervening heating period of not less than 10 minutes, which shall show a further loss in weight of not more

than 0.1 per cent. Calculate the weight of bituminous saturation by difference. The saturant may be recovered by combining the extract and washings in a 200-ml. distilling flask, provided with a dropping funnel to add the solution during distillation, to which is attached a water-cooled condenser and a receiver, the latter having a vented connection to a vacuum pump. The distilling bulb is immersed in a bath having a temperature of about 212°F. (100°C.) if the solvent were carbon disulfide and a temperature of about 275°F. (135°C.) if benzol or chloroform were used as the solvent. Distillation shall be continued at this temperature at atmospheric pressure until no more solvent is evolved, after which vacuum shall be applied very gradually until a vacuum of 0.5 in. mercury pressure is attained. Great care shall be exercised in applying the vacuum gradually to prevent foaming. This vacuum shall be continued for one hour after the specified vacuum has been reached. The distilling flask shall then be disconnected and the residue represents the felt saturant and shall be weighed.

NOTE.—Use the residue of bituminous saturation recovered from the felt for examining its physical and chemical characteristics.

Calculate the per cent of bituminous saturation (B-3) carried by the dry felt. (5)

Calculate the weight in pounds per 108 sq. ft. of bituminous saturation (B-3) present in the dry felt, i.e., $(5) \times (3)$ (6)

Method (II): The following method of test has been standardized for determining the paraffin content of waxed papers:²⁸

This method of test covers the procedure for determining quantitatively the amount of paraffin in wax-impregnated (waxed) paper.

NOTE.—The scope of this method is not intended to include paraffin-sized papers. The extract from this type of paper generally contains such quantities of soluble matter other than waxes that the accuracy of the method for small amounts of paraffin is questionable.

The apparatus shall consist of the following:

(a) Extractor: Soxhlet or Underwriters' Laboratories extraction apparatus.

(b) Funnels: Two 250-ml. separatory funnels.

(c) Carbon Tetrachloride, C.P.

(*d*) Petroleum Ether, C.P.

(*e*) Alcoholic Potassium Hydroxide (approximately 0.5 *N*).

The test specimen shall be cut from the sample in such a way as to be thoroughly representative. It shall consist of not less than 3 g. of the paper, in the as-received condition, cut into strips approximately 0.5 in. in width and folded into numerous small cross-wise folds (Note).

NOTE.—The folding is essential to secure complete and quick extraction. Do not tear the specimen into small pieces, since they will stick together and the paper may not be completely extracted.

Place the prepared test specimen in the extractor, and add carbon tetrachloride (CCl_4). If a Soxhlet extractor is used, the strips of paper should be below the surface of the CCl_4 when the siphon cup is filled. Extract the specimen until the wax is all removed, collecting the extract in a flask (the extraction generally requires at least 6 hrs.). This is not necessary with the Underwriters' Laboratories extractor, where the strips are surrounded by the hot vapors. In either case, enough solvent shall be added to cause the siphon to run over, and then an additional quantity to fill the cup one-half to two-thirds full.

Evaporate the extract on a water bath to dryness, add 25 ml. of the 0.5 *N* alcoholic KOH, and again evaporate to dryness. Cool the dry residue, take it up with petroleum ether and water, and transfer to a separatory funnel. The volume of ether in the funnel shall be about 25 ml. and that of the water 150 ml. The water shall contain a small amount of NaCl to prevent emulsification. Shake the contents of the flask thoroughly, let the two layers separate completely, and draw off the water into a second separatory funnel. Re-extract the water layer one or more times, as may be found necessary, with a fresh 25-ml. portion of petroleum ether. Combine the ether extracts and wash with fresh 100-ml. portions of water until the separated liquids are perfectly clear. An addition of a strong solution of NaCl may be found necessary.

Transfer the petroleum ether extract to a weighed evaporating dish or flask, evaporate to dryness, dry at 100 to 105° C. for 1 hr., and weigh. The weight so found shall be taken to represent the paraffin in the specimen (Note).

NOTE.—The procedure is based on the assumption that all unsaponifiable material soluble in petroleum ether will be paraffin. With the development and growing use of new materials in waxed paper, it may no longer be assumed to contain only paraffin, as there may be present higher alcohols, either free or resulting from saponification of esters or other materials not paraffin, which are soluble in petroleum ether. However, the possible error resulting from these materials is small and may be considered negligible.

The amount of paraffin shall be reported as a percentage of the original weight of the waxed paper. Not less than two determinations shall be made, and the average result rounded off to the nearest 0.1 per cent shall be reported.

Duplicate determinations should agree within 0.2 per cent.

Bituminous Matter in the Top and Bottom Coatings Respectively (B-1 and B-2): The horizontal sections of the weather side (which may be either side on type X roofing) containing the asphalt coating and mineral surfacing shall now be extracted with carbon disulfide in a suitable extractor until the washings are colorless. The insoluble material shall be removed and dried in air and the pieces of felt picked out and brushed free of adhering mineral matter. This felt shall then be placed in a tared weighing bottle, further dried at 105 to 110° C. (221 to 230° F.) for 30 min., and weighed. From this weight corrected for excess ash and the percentage of asphalt saturant determined, the weight of asphalt in this felt shall be calculated. The mineral matter shall be dried in air until free from carbon disulfide, then placed in a tared weighing bottle, further dried for 30 min. at 105 to 110° C. (221 to 230° F.), cooled in a desiccator, and weighed. This mineral matter shall be retained for sieve analysis.

Calculation: From the original weight of the sample subtract the combined weights of felt, asphalt, saturant in the felt, and mineral matter. The difference is the weight of weather side coating (soluble in carbon disulfide) per 108 sq. ft.

The horizontal sections of the reverse side (which on Type X roofing will be the side opposite to that selected) containing the coating and mineral surfacing shall now be extracted with carbon disulfide and the weight of coating soluble in carbon disulfide calculated. The mineral matter recovered shall be combined with that obtained from the weather side and retained for sieve analysis. The felt shall be ashed to determine the true weight of felt.

Calculate the weight in pounds per 108 sq. ft. of bituminous matter (B-1) in the top coating. (7a)

Calculate the weight in pounds per 108 sq. ft. of bituminous matter (B-2) in the bottom coating. (7b)

Calculate the weight in pounds per 108 sq. ft. of total mineral matter embedded and admixed with the top coating. (8a)

Calculate the weight in pounds per 108 sq. ft. of total mineral matter embedded and admixed with the bottom coating. (8b)

The following modified procedure may be used to separate the bituminous constituents in the coatings for further examination and also to check the percentage of mineral matter, both detached and embedded in the top and bottom coatings: The detached mineral matter shall be brushed off from six weighed 2-in. by 8-in. strips; the strips shall again be weighed and the percentage calculated based on the original weight of strips. The outer portion of the top and bottom surface coatings, respectively, shall then be scraped off after the strips have been heated in an oven at 212° F. (100° C.) for 30 minutes. The coatings shall be scraped off by holding a dull knife at right angles to the strip of roofing supported on a firm level surface and drawing the blade sideways under moderate pressure. Care shall be taken to avoid scraping entirely through the surface coating. This is important. The scrapings shall then be weighed and dissolved in benzol (boiling-point 80 to 82° C.) or C.P. carbon disulfide; and the mineral matter separated by filtering or centrifuging and washing with successive portions of solvent. The mineral matter shall then be dried at 225° F. (107° C.), cooled in a desiccator and weighed. Calculate the percentage of mineral matter based on the original weight of roofing strips taken, which should check with that obtained previously. The bituminous matter in the scrapings shall be calculated by difference and reduced to a percentage basis on the original roofing strips used. The bituminous coating shall be recovered by evaporating off the solvent as described. Both surface coatings should be treated separately in this manner.

NOTE.—Use the separated bituminous coatings for examining their physical and chemical characteristics.

Coarse or Moderately Coarse Embedded Mineral Matter (A-2), also Fine Admixed Mineral Matter (A-4) on the Top Coating of Types Y and Z: The mineral matter recovered from the top sections of Types Y and Z shall be boiled with 100 ml. water and washed through a 65-mesh copper wire screen with sufficient water to remove the fine mineral matter. The total aqueous suspension of mineral matter which passes through the screen shall then be recovered by filtering the washings through a weighed Gooch crucible, then dried at 225° F. (107° C.) and reweighed. This represents the fine mineral matter admixed with the top coating. The mineral constituents retained on the 65-mesh screen represent the coarse or moderately coarse mineral matter embedded in the top coating. This shall likewise be dried and weighed.

Calculate the weight in pounds per 108 sq. ft. of coarse or moderately coarse mineral matter (A-2) embedded in the top coating of Types Y and Z. (9)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter (A-4) admixed with the top coating of Types Y and Z (*) (10)

Fine Embedded and Admixed Mineral Matter (A-2 and A-4) in the Top Coating of Type X; Likewise Fine Embedded and Admixed Mineral Matter (A-3 and A-4) in the Bottom Coating of Types X and Y. Take five of the 2-in. by 8-in. strips, from which the detached mineral matter has been washed off with water, and scrape off the *outer* layer of the top and bottom coatings, respectively, by means of moderately rough sand paper or a knife blade. Enough of the surface shall be scraped to remove every vestige of the fine embedded mineral matter, and at the same time care should be taken not to cut completely through the surface coatings into the saturated felt underneath. Then scrape off about 5.0 g. of the top and bottom surface coatings, respectively, with a sharp knife as described, taking care not to scrape entirely through the surface coatings. Ignite the 5.0-g. portions separately in a tared crucible until all the carbon has been consumed, add a few drops of ammonium carbonate solution, ignite to a dull red heat and weigh.

* Up to 2 per cent admixed mineral matter probably adventitious and not filler.

Calculate the *percentage* by weight of fine mineral matter admixed with the top coating (Type X)..... (11a)

Calculate the *percentage* by weight of fine mineral matter admixed with the bottom coating (Types X and Y)..... (11b)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter admixed with the top coating (Type X), i.e., $(7a) \times (11a)$ (12a)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter admixed with the bottom coating (Types X and Y), i.e., $(7b) \times (11b)$ (12b)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter embedded in the top coating (Type X), i.e., $(8a) - (12a)$ (13a)

Calculate the weight in pounds per 108 sq. ft. of fine mineral matter embedded in the bottom coating (Types X and Y), i.e., $(8b) - (12b)$ (13b)

The total mineral matter recovered plus the excess ash shall be moistened with a few drops of alcohol, boiled with 100 ml. of water, and washed successively through No. 6 (3360-micron), and No. 100 (149-micron) sieves with sufficient hot water to remove the fine mineral matter. The mineral matter retained on each sieve shall be collected separately, dried at 105 to 110° C. (221 to 230° F.) for 30 min., and weighed. From these weights, the weight of mineral matter shall be calculated and reported as follows:

Passing Sieve	Retained on Sieve	Weight of Mineral Matter per 108 Sq. Ft.
No. 6 (3360-micron)	No. 100 (149-micron)
No. 100 (149-micron) ^a

^a By difference.

To check the weight of mineral matter passing a No. 100 (149-micron) sieve, the total aqueous suspension of mineral matter which passes through the No. 100 sieve shall be recovered by fil-

tering through a weighed Gooch crucible, dried at 105 to 110° C. (221 to 230° F.), and weighed.

The following modified procedure should be used for the analysis of heavy-butt individual or strip-shingles:

Specimens 2 in. in width and 8 in. in length shall be cut from the thin portion of samples selected. These specimens shall be analyzed in accordance with the procedures described, except that the calculation of the weight of the weather side coating shall be made by subtracting from the original weight of the sample the combined weight of the felt, the saturant in the felt, and the mineral matter passing a No. 6 (3360-micron) sieve and retained on a No. 100 (149-micron) sieve. [It is assumed that all granular surfacing and the greater portion of talc surfacing will be retained on a No. 100 (149-micron) sieve, and that the mineral filler will pass through.]

Specimens from the thick portion of the shingle shall be secured and analyzed in accordance with the procedure described.

The composition of the shingle as a whole, with reference to the specified requirements, shall be calculated from the combined areas of the respective portions analyzed.

NOTE.—The method of analysis described in this section is not suitable for analyzing thick butt shingles of the "Tapered Type."

Compilation of Results: The foregoing items shall be reported in pounds per 108 sq. ft. as shown in Table CLIII. If desired, the results may also be expressed in per cent by weight.

(C) RECOVERY AND EXAMINATION OF EXTRACTED COATINGS AND SATURATION

The bituminous constituents separated in Test 76 by extraction shall be recovered as described in Test 21*b*, whereupon they may be examined further by appropriate tests given in Chapter XXXII.

(D) EXAMINATION OF THE SEPARATED MINERAL SURFACING AND ADMIXED MINERAL CONSTITUENTS

Test 76*a*. Sieve Analysis of Granular Mineral Surfacing. The following method of test has been standardized for the sieve

TABLE CLIII

ANALYTICAL DATA PERTAINING TO SATURATED AND COATED FABRICS

	Type X	Type Y	Type Z
<i>Mineral Matter:</i>			
Fine mineral matter embedded in top coating.....	Item (13a)
Coarse or moderately coarse mineral matter embedded in top coating.....	Item (9)	Item (9)
Fine mineral matter embedded in bottom coating.....	Item (13b)	Item (13b)
Fine mineral matter admixed with top coating....	Item (12a)	Item (10)	Item (10)
Fine mineral matter admixed with bottom coating.....	Item (12b)	Item (12b)
<i>Bituminous Matter:</i>			
Contained in top coating.....	Item (7a)	Item (7a)	Item (7a)
Contained in bottom coating.....	Item (7b)	Item (7b)
Saturant of the felt.....	Item (6)	Item (6)	Item (6)
<i>Fibrous Matter:</i>			
Felt after desaturation.....	Item (3)	Item (3)	Item (3)
Net weight of roofing material.....	Sum	Sum	Sum
Check total.....	Item (1)	Item (1)	Item (1)
Detached mineral matter.....	Item (2)	Item (2)	Item (2)
Packing material, nails and cement.....	Weight	Weight	Weight
Gross weight per 108 sq. ft.....	Sum	Sum	Sum
<i>Per Cent Ash:</i>			
From desaturated felt.....	Item (4)	Item (4)	Item (4)

analysis of granular mineral surfacing for use in manufacturing asphalt roofing and shingles: ²⁴

This method is intended for the sieve analysis of granular mineral surfacing material, such as crushed slate, stone, etc., used on the weather surface of prepared asphalt roofing and shingles.

(a) Sieves: A set of consecutive sieves of the series listed in Table CLIV, conforming to Specifications A.S.T.M. Designation: E 11 shall be used. The group of sieves selected shall include those appropriate to the grading of the granular mineral to be subjected to sieve analysis. Sieves of either the coarser or finer openings, on which less than 0.05 per cent of the weight of the sample of the particular material would be found after sieving, need not be included in the set. The wire cloth for these sieves shall be woven (not twilled) from brass, bronze, or other suitable wire, and shall be mounted without distortion in circular frames, 8 in. (20.32 cm.) in diameter and about 2 in. (5 cm.) between the top of the frame and the cloth.

TABLE CLIV

NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES

Size or Sieve Designation	Sieve Opening		Permissible Variation in Average Opening, per cent	Permissible Variation in Maximum Opening, per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
3360 micron (No. 6)	3.36	0.1320	±3	+10	0.87 to 1.32	0.034 to 0.052
2380 micron (No. 8)	2.38	0.0937	±3	+10	0.74 to 1.10	0.0291 to 0.0433
1680 micron (No. 12)	1.68	0.0661	±3	+10	0.62 to 0.90	0.0244 to 0.0354
1190 micron (No. 16)	1.19	0.0469	±3	+10	0.50 to 0.70	0.0197 to 0.0276
840 micron (No. 20)	0.84	0.0331	±5	+15 *	0.38 to 0.55	0.0150 to 0.0217
590 micron (No. 30)	0.59	0.0232	±5	+15 *	0.29 to 0.42	0.0114 to 0.0165
420 micron (No. 40)	0.42	0.0165	±5	+25 *	0.23 to 0.33	0.0091 to 0.0130
297 micron (No. 50)	0.297	0.0117	±5	+25 *	0.170 to 0.253	0.0067 to 0.0100
210 micron (No. 70)	0.210	0.0083	±5	+25 *	0.130 to 0.187	0.0051 to 0.0074
149 micron (No. 100)	0.149	0.0059	±6	+40 *	0.096 to 0.125	0.0038 to 0.0049

* Not more than 5 per cent of the openings shall exceed the nominal opening by more than one half of the permissible variation in maximum opening.

(b) Sieve Shaker: A mechanically operated sieve shaker, which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete.

(c) Sample Splitter: A riffle sampler with $\frac{3}{8}$ - or $\frac{1}{2}$ -in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

Each carload of mineral granules shall be considered the unit for sampling, except that where a carload includes more than one kind of granular surfacing, the entire quantity of each kind in the car shall be considered the unit for each kind, respectively.

In collecting the gross sample from a carload of mineral granules shipped in bulk, eight equal increments of not less than 2 lbs. each shall be taken from the material at the exposed surface of the car. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sample taken from the bottom of the excavation. The eight sampling points shall be located as shown in Fig. 373.

In collecting the gross sample from a carload of mineral gran-

ules shipped in bags, a number of bags shall be selected at random, equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 2 lbs. from each of the bags taken for sampling.

Where the mineral granules are being loaded or unloaded by conveyor or chute, the gross sample shall preferably be collected by taking equal increments from the stream at regular time intervals, with such frequency that the total weight of the gross sample will be not less than 16 lbs. Increments shall be taken from the full width and thickness of the stream, preferably, if practicable, by inserting a suitable container into the stream.

The gross sample shall be reduced by riffing or hand-quartering to not less than 500 g. (1.1 lbs.).

The entire sample obtained from reduction of the gross sample, shall be weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

The group of sieves selected from the series shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed, shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device.

The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 15 min. Since granular mineral surfacing materials usually have been subjected to grading by screens in the process of manufacture and hence separate rapidly into their sieve fractions, an end point determination is not considered necessary.

The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

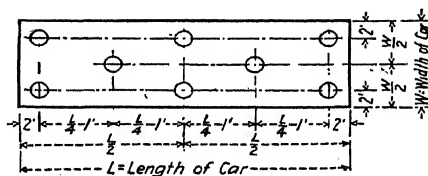


FIG. 373.—Location of Sampling Points from Surface of Car.

The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

Retained on Sieve	Passing Sieve	Per Cent
3360-micron (No. 6).....
2380-micron (No. 8).....	3360-micron (No. 6).....
1680-micron (No. 12).....	2380-micron (No. 8).....
1190-micron (No. 16).....	1680-micron (No. 12).....
840-micron (No. 20).....	1190-micron (No. 16).....
590-micron (No. 30).....	840-micron (No. 20).....
420-micron (No. 40).....	590-micron (No. 30).....
297-micron (No. 50).....	420-micron (No. 40).....
210-micron (No. 70).....	297-micron (No. 50).....
149-micron (No. 100).....	210-micron (No. 70).....
	149-micron (No. 100).....
Total.....

The sum of the sieve weight percentage fractions, reported as above tabulated, shall be not less than 99.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 0.5 per cent.

Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

Test 76b. Sieve Analysis of Nongranular Mineral Surfacing. The following method of test has been standardized for the sieve analysis of nongranular mineral surfacing for use in manufacturing asphalt roofing and shingles:²⁵

This method is intended for the sieve analysis of nongranular mineral surfacing material, such as mica, talc and other powdered or flaky mineral particles, used on the weather surfaces of prepared asphalt roofing and on the non-weather-exposed surface of asphalt shingles.

(a) **Sieves:** A set of consecutive sieves of the series listed in Table CLV, conforming to Specifications A.S.T.M. Designation: E 111 shall be used. The group of sieves selected shall include those appropriate to the grading of the nongranular mineral to be subjected to sieve analysis. Sieves of either the coarser or finer openings, on which less than 0.05 per cent of the weight of the sample

TABLE CLV

NOMINAL DIMENSIONS, PERMISSIBLE VARIATIONS, AND LIMITS FOR WOVEN WIRE CLOTH OF STANDARD SIEVES

Size or Sieve Designation	Sieve Opening		Permissible Variation in Average Opening, per cent	Permissible Variation in Maximum Opening, per cent	Wire Diameter	
	mm.	in. (approx. equivalents)			mm.	in. (approx. equivalents)
1680 micron (No. 12)	1.68	0.0661	±3	+10	0.62 to 0.90	0.0244 to 0.0354
1190 micron (No. 16)	1.19	0.0469	±3	+10	0.50 to 0.70	0.0197 to 0.0276
840 micron (No. 20)	0.84	0.0331	±5	+15 *	0.38 to 0.55	0.0150 to 0.0217
590 micron (No. 30)	0.59	0.0232	±5	+15 *	0.29 to 0.42	0.0114 to 0.0165
420 micron (No. 40)	0.42	0.0165	±5	+25 *	0.23 to 0.33	0.0091 to 0.0130
297 micron (No. 50)	0.297	0.0117	±5	+25 *	0.170 to 0.253	0.0067 to 0.0100
210 micron (No. 70)	0.210	0.0083	±5	+25 *	0.130 to 0.187	0.0051 to 0.0074
149 micron (No. 100)	0.149	0.0059	±6	+40 *	0.096 to 0.125	0.0038 to 0.0049
74 micron (No. 200)	0.074	0.0029	±7	+60 *	0.045 to 0.061	0.0018 to 0.0024

* Not more than 5 per cent of the openings shall exceed the nominal opening by more than one half of the permissible variation in maximum opening.

of the particular material would be found after sieving, need not be included in the set (Note). The wire cloth for these sieves shall be woven (not twilled) from brass, bronze, or other suitable wire, and shall be mounted without distortion in circular frames, 8 in. (20.32 cm.) in diameter and about 2 in. (5 cm.) between the top of the frame and the cloth.

NOTE.—For relatively coarse or flaky materials, such as coarse mica, sieves from 1190 to 210-micron (Nos. 16 to 70), inclusive, will usually be found suitable; for finer surfacing materials, such as fine mica or talc flour, sieves from 590 to 74-micron (Nos. 30 to 200), inclusive, will usually give a satisfactory sieve analysis.

(b) Sieve Shaker: A mechanically operated sieve shaker, which imparts to the set of sieves a rotary motion and tapping action of uniform speed, shall be used. The number of taps per minute shall be between 140 and 160. The sieve shaker shall be fitted with a hard maple plug to receive the impact of the tapping device. The entire apparatus shall be rigidly mounted by bolting to a solid foundation, preferably of concrete.

(c) Sample Splitter: A riffle sampler with $\frac{3}{8}$ - or $\frac{1}{2}$ -in. divisions, for reducing the gross sample to the quantity required for the sieve analysis, shall be used.

Each carload of nongranular mineral surfacing shall be considered the unit for sampling.

In collecting the gross sample from a carload of nongranular

mineral surfacing shipped in bags, a number of bags shall be selected at random equivalent to the cube root of the total number of bags in the car. The gross sample shall consist of equal increments of not less than 0.5 lb. from each of the bags taken for sampling.

In collecting the gross sample from a carload of nongranular mineral surfacing material shipped in bulk, at least eight equal increments of not less than 0.5 lb. each shall be taken from well distributed points in the mass, using a sampling tube not less than 1 in. in diameter. A conical excavation about 1 ft. in depth shall be made at each sampling point and the sampling tube inserted into the mass at the bottom of the excavation.

The gross sample shall be reduced by riffing or hand-quartering to not less than 100 g. (0.22 lb.).

The entire sample obtained from reduction of the gross sample, shall be dried at least 2 hr. in an oven at 220° F., weighed with an accuracy of not less than 0.1 g., and taken for the sieve analysis.

The group of sieves selected shall be assembled in consecutive order as to size of openings, with the sieve having the largest openings at the top and the one with the smallest openings at the bottom, the assembly being completed by a solid collecting pan below the bottom sieve. The test sample, previously weighed, shall be placed on the top sieve and this sieve closed with a solid cover. The sieve assembly shall then be securely fastened in a suitable mechanical sieve shaking device.

The sample shall be passed through the sieves of the series selected, by subjecting it to the action of the sieve shaker for a period of 20 min. At the end of this period the collecting pan, containing the portion of the material passing the finest sieve of the group selected, shall be removed from the sieve assembly and the contents weighed with an accuracy of not less than 0.1 g. The collecting pan shall then be re-assembled with the sieves, as before, and the shaking continued for an additional 10 min. At the end of this additional shaking period the collecting pan shall be removed and the contents weighed. If the additional material passing the finest sieve during this second shaking period does not exceed 0.5 per cent of the total weight of the sample, the sieve analysis shall be considered complete. If it does exceed 0.5 per cent, the collecting pan and sieves shall be re-assembled and shaken for successive addi-

tional 10-min. periods, weighing the material collected in the pan after each period of shaking, until the amount passing the finest sieve in a 10-min. shaking period is less than 0.5 per cent of the weight of the sample.

The portion of the sample retained on each of the sieves and on the pan shall be carefully removed and weighed with an accuracy of not less than 0.1 g.

The results of the sieve analysis shall be reported to the nearest 0.1 per cent (omitting the results on those sieves on which less than 0.05 per cent of the total weight of the test sample was collected), as follows:

Retained on Sieve	Passing Sieve	Per Cent
1680-micron (No. 12)	1680-micron (No. 12)
1190-micron (No. 16)	1190-micron (No. 16)
840-micron (No. 20)	840-micron (No. 20)
590-micron (No. 30)	590-micron (No. 30)
420-micron (No. 40)	420-micron (No. 40)
297-micron (No. 50)	297-micron (No. 50)
210-micron (No. 70)	210-micron (No. 70)
149-micron (No. 100)	149-micron (No. 100)
74-micron (No. 200)	74-micron (No. 200)
Total

The sum of the sieve weight percentage fractions, reported as above tabulated, shall be not less than 98.5 per cent, that is, the total loss and error in the sieve analysis shall not exceed 1.5 per cent.

Duplicate determinations by the same operator, using the same sieves, shall check within 1 per cent of the total weight of the sample, for the portion of the sample retained on each sieve and on the pan.

Mineral granules may be subjected to the following additional tests: ²⁸

Test 76c. Solubility and Color Fixation. Wash a sufficient amount of granules with benzol to remove any oils or any other treating material that may be on the granules. Weigh (on the analytical balance) 20 gm. of the washed granules, after they have been dried, into a dry 500 ml. Erlenmeyer flask. Add 100 ml. of distilled water, connect with a reflux condenser and boil on the hot plate for 2 hrs. At the end of this time disconnect the flask from the condenser and decant the solution into a 400-ml. beaker. Care

is to be observed that no granules are permitted to be carried over into the beaker. Wash three times, using about 50 ml. of hot distilled water for each washing, decanting the wash water into the same beaker with the solution. The final wash should come from the granules clear and free from any visible pigment or any soluble matter. This beaker containing the extract and washings is to be set aside for further examination. The granules are now washed out of the flask into a suitable receptacle and dried in the oven at 105°C . They are again weighed on the analytical balance. The loss in weight is calculated to percentage and is reported as percentage of total material removed.

The extract and washings in the beaker are filtered through a weighed Gooch crucible containing a close asbestos mat. If the filtrate is not clear, it is to be filtered again through the same crucible so that all pigment and insoluble matter are collected for weighing. Dry the crucible and contents at 105°C . in the oven and weigh. The increase in weight is reported in percentage of the original weight of the sample as the insoluble matter removed.

Test 76d. Color Stability. This test is performed as follows:

(a) Leaching: Approximately 25 g. of the granules, previously washed in petroleum naphtha and dried, shall be washed free of dust with freshly distilled water and placed in a 100-cc. pyrex or hard glass beaker. Cover the granules to a depth of $\frac{1}{8}$ in. with freshly distilled water and place in oven at 150°F . for 24 hrs. This to be repeated every 24 hrs. for 5 days.

The granules after this treatment should show no undesirable change in color.

(b) Sulfide Discoloration: (The effect of hydrogen sulfide, H_2S , in the atmosphere.) A sample of granules (25–50 g.) is placed in an upright glass tube and covered with a $N/100$ sulfuric acid solution. The hydrogen sulfide generator is then started. A slight vacuum is next applied to the system. This draws the air into the gas generator, mixes it with the H_2S gas being generated, draws the mixture through a gas trap, bubbles it up through the granules and finally exhausts the gas out through the top of the tube. The $N/100$ H_2SO_4 solution covering the granules quickly becomes saturated with H_2S . After 30 min., the sample is removed, washed, dried and its color compared with that of the original sample.

This test indicates the presence in the color coating of the granules of any substance which forms dark colored compounds by reaction with hydrogen sulfide.

Test 76e. Blooming (Efflorescence). Approximately 25 g. of granules, previously washed with petroleum naphtha and dried, are distributed over the bottom of a petri dish of 10 cm. diameter, and kept moistened with a 5–10 cc. distilled water for 48 hrs.

Place the uncovered petri dish and sample in a refrigerator, the temperature of which is maintained from 35 to 50° F. and a current of air circulated over the sample by means of a small electric fan, until the moisture has been evaporated from the granules under test.

The presence of constituents of the granules that may cause undesirable blooming when the roofing is placed in service will be indicated by this test.

Test 76f. Affinity for Asphalt (Adhesion). The test panel is examined under the low power of a Bausch & Lomb AKW-S Wide Field Binocular Microscope. This microscope has the advantage of a wide field under low powers and an excellent depth of focus. The examination is made by testing the adhesion of individual particles, using a No. 6 Single End Dental Explorer. By means of this needle which has a satisfactory tip, the individual granules are picked carefully from the surface of the roofing, and examined as they become loose, for the presence of asphalt coating.

The characteristic appearance of granules with adhering asphalt as removed by this method from the roofing, as well as granules which show no adhering asphalt, is apparent.

Using the low power of the microscope, ten granules are removed from a field and examined individually for presence of asphalt from the coating. In case the granules show adhering asphalt they are marked plus; if no asphalt is observed, they are recorded minus. Repeat this procedure on nine additional fields selected at random over the surface of the roofing. After ten fields have been examined and the results recorded, total the number of plus granules and record this figure as the adhesion value of the material. The accuracy of the method may be increased by examining a large number of fields, or more granules in each field, and calculating the plus granules to percentage.

Test 76g. Staining. Certain natural and artificially colored granules have a very porous surface. On roofing made with these granules, especially when the asphalt coating contains a relatively high proportion of volatiles, the surfacing is prone to absorb the more volatile constituents and become extremely dark in color. This absorption is cumulative and once the reaction has taken place, the granules give up this absorbed material with great reluctance. All absorbent granules stain to a greater or lesser degree, depending upon the amount of pore space and the physical structure of the granules. In the case of natural slate and greenstone, prior to the introduction of artificially colored granules, this question of staining was of considerable moment.

Test 76h. Susceptibility to Atmospheric Dirt (Sooting). This test is designed to give some measure of the tendency of roofing granules to darken or "soot" on exposure. The procedure is as follows: 25 g. of granules are shaken in a benzene suspension of carbon black, consisting of 1 g. of carbon black in 100 cc. of benzene, for a period of 2 min., allowed to stand 2 min., shaken 2 min. more, and then decanted. Following this, they are rinsed four times with 50 cc. of benzene and then dried. After being dried, the granules are compared with the original sample and the degree of sooting noted being expressed as trace, slight, moderate, severe, or excessive.

(E) EXAMINATION OF THE SEPARATED PAPER, FELT, OR TEXTILE

The fabric separated upon extraction in Test 76 may be subjected to the following tests:

Test 77. Weight per Unit Area ("Number"). This is expressed arbitrarily by the trade in terms of the so-called "number."

(I) *For Paper*, the "number" usually corresponds to the weight in pounds of a ream, consisting of 500 sheets, each sheet measuring 25 in. by 40 in. Other methods are also sometimes used embodying a different number of sheets, or sheets of other sizes.

The following method of test has been standardized for determining the basis of weight of paper and paper products:²⁷

This method of test covers the procedure for determining the basis weight of large and small pieces of paper. Factors for con-

version of basis weight from one commercial size of paper to another are given below.

The apparatus shall consist of the following:

(a) Balance: A balance having a sensitivity of not less than 0.25 per cent of the load applied and so graduated that readings of this degree of accuracy can be made. The balance should preferably be a specially constructed sheet-weighing device indicating the equivalent weight of a 500-sheet and also a 480-sheet ream in pounds, when a specimen consisting of one sheet of the designated size is weighed. The balance shall be protected from air currents while weighing.

(b) Measuring Device: A device capable of measuring the size of the specimen to an accuracy within 0.25 per cent of the smallest dimension to be measured.

(c) Paper Cutter: A template for cutting the paper to size. When a template is used for preparing the sheets, it is recommended that the paper be cut to exact size with a sharp knife. A paper cutter having an attachment for ensuring parallelism of the opposite edges is also recommended.

The balance shall be level at all times and care shall be taken that the level is not disturbed. The balance shall be calibrated after leveling by applying accurate weights with increasing and decreasing loads. If the level of the balance is not disturbed, the frequency of calibration depends entirely upon the condition and frequency of use and, therefore, becomes largely a matter of judgment.

The test specimens shall consist, whenever possible, of at least 10 sheets each approximately 100 sq. in. (645 sq. cm.) in area taken from the sample obtained as prescribed.

All test specimens shall be brought to a standard condition, prior to testing, in accordance with A.S.T.M. Designation: D 685. All tests shall be made under the standard atmospheric conditions.

The area of the test specimen shall be determined to the nearest 0.25 per cent of its total area. The weight of the specimen shall be determined to the nearest 0.25 per cent of its total weight. If weighed in grams, the weight of 10 sheets multiplied by 1.102 gives the equivalent weight in pounds of 500 sheets of the same area.

The report shall include at least two of the following items:

- (a) Equivalent ream weight in pounds for a ream consisting of 500 sheets 25 by 40 in. (635 by 1016 mm.) in size,
 (b) Weight in grams per square meter of paper, and
 (c) Equivalent weight for the ream size commonly used by the paper industry for the particular type of paper.

The weight shall be reported to three significant figures. If less than 1000 sq. in. (0.6 sq. m.) of paper is used for the test, the area tested shall be reported.

Duplicate determinations made on the same sample shall agree within 2 per cent.

The information given below will be of assistance in calculating the weight of paper and the conversion of customary trade-sizes 500-sheet reams, to and from the weight in pounds of size 25 by 40, and to the weight in grams per square meter.

When specimen sheets are weighed on a balance in grams or on a paper scale, the weight of trade size 25 by 40, 500-sheet ream is calculated as follows:

$$P = \frac{1102g}{abn} \quad \text{or} \quad \frac{7111g}{yzn}$$

$$P = \frac{1000p}{abn} \quad \text{or} \quad \frac{6451p}{yzn}$$

$$G = \frac{1550g}{abn} \quad \text{or} \quad \frac{10,000g}{yzn}$$

$$G = \frac{1406p}{abn} \quad \text{or} \quad \frac{9070p}{yzn}$$

where P = weight in pounds of trade size 25 by 40, 500-sheet ream,
 G = weight in grams per square meter of trade size 25 by 40,
 500-sheet ream,

a = length of specimen in inches, or

y = length of specimen in centimeters,

b = width of specimen in inches, or

z = width of specimen in centimeters,

g = weight in grams of sheets weighed, or

p = indicated weight on scale for 500 sheet ream, and

n = number of sheets weighed.

In stating the "number," the percentage of moisture present in the paper should also be given.

Moisture Content of Paper: The following procedure has been proposed²⁸ for ascertaining the moisture content of paper intended for electrical insulation purposes:

A weighing bottle, evaporating dish, thermometer, constant-temperature oven, chemical balance and desiccator are necessary for the test. The weighing bottle should be of convenient size, about 65 mm. (2.56 in.) in height and 45 mm. (1.77 in.) in diameter, with a wide mouth provided with a ground-glass stopper. The chemical balance should be sensitive to 0.1 mg.

The moisture content figure is used to calculate the percentage of acidity, size, and ash of the paper to the bone-dry basis. It shall be determined on 2- to 5-g. samples.

If the moisture content of the paper as received at the purchaser's works is desired or at any other stated time, it shall be determined on the specimen taken at the definitely stated time by cutting small pieces of approximately 6.5 sq. cm. (1 sq. in.) in area from each of the required samples and immediately placed into an air-tight container.

NOTE.—Since paper adjusts itself, as regards moisture content, to the humidity of the surrounding atmosphere in a very short time (2 or 3 min.), special care must be exercised to transfer rapidly the sample from the roll or bundle to the container and from the container to the weighing bottle.

The test specimen before drying shall be weighed in the tared bottle with the stopper in place. The bottle shall then be placed in the oven at 100 to 105° C. (212 to 221° F.), the stopper removed and laid alongside of the bottle and the contents transferred to the drying dish. After one hour, while still in the oven, the sample shall be replaced in the weighing bottle, and the bottle stoppered and transferred to the desiccator. The stopper should be removed while the bottle cools. When the specimen and the bottle have cooled to room temperature, the stopper shall be replaced and the bottle with its contents weighed. This process shall be repeated at intervals of one hour until the difference in weight between two successive weighings is not more than 0.2 per cent of the weight of the specimen.

NOTE.—The weighing bottle or sample should not be touched with the fingers during this test.

The moisture content shall be expressed:

- (a) as a percentage of the weight of the dry sample, and
- (b) as a percentage of the weight of the undried sample.

Weight of Conditioned Paper: Samples shall be conditioned in air maintained at a relative humidity between 60 and 65 per cent as measured with a sling psychrometer or its equivalent. The temperature of the air shall be maintained as constant as possible at some temperature between the limits of 20 and 30° C. (68 and 86° F.). The samples should remain in the conditioned air for not less than 4 hours prior to the tests and should be supported so as to allow a free circulation around each sample.

Each specimen while in the conditioned atmosphere shall be cut accurately to any convenient size and accurately weighed. Specimens of sufficient size to weigh at least from 3 to 5 g. (0.11 to 0.18 oz.) should be taken. If it is not possible to do the weighing in the conditioned atmosphere, the specimen shall be placed in a weighing bottle and tightly stoppered with a ground-glass stopper before being removed from the conditioned atmosphere. The weight shall be calculated to and expressed as grams per square meter.

NOTE.—Grams per square meter times 0.00142 equals pounds per 1000 sq. in., from which the weight in pounds per ream of any size can be calculated if desired.

Methods have also been standardized for ascertaining the acidity or alkalinity of paper;²⁹ likewise the resin content of paper.³⁰

(II) *For Felt:* The "number" represents the weight in pounds of a ream consisting of 480 sheets, each measuring 12 in. × 12 in., the moisture content of which is arbitrarily set at 3.5 per cent. The following formula may be used (corrected for any carbonaceous matter present, as ascertained in Test 75).

$$\text{"Number"} (480 : 12 \times 12) = \frac{\text{Gms.}}{\text{Sq. cms.}} \times 983 = \frac{\text{Gms.}}{\text{Sq. ins.}} \times 152$$

Uncorrected "Number of Felt": The "uncorrected number" represents the weight of the felt as received in pounds per 480 sq. ft. To ascertain the "uncorrected number," unwind the first ten convolutions of each roll sampled, and with a knife and straight-edge cut cleanly across the sheet at right angles to the sides. Re-

move a section measuring 24 in. in the direction of the roll's length. From this, with a knife and template, cut three specimens, each measuring a "square foot," one from the center, and one close to each edge of the sheet. A "square foot" shall imply 144 sq. in. within that degree of accuracy which will not affect the weight thereof on a paper makers' scale more or less than $\frac{1}{2}$ lb. per 480 sq. ft., from 1 sq. ft. area absolute. Rapidly weigh each square foot specimen and compute the "number" (i.e., the weight in pounds per 480 sq. ft.). Average the three readings for each roll sampled, and from these results, compute the minimum, maximum, and average "uncorrected number" of the felt. Unless the square foot samples are tested immediately by the laboratory, they should be preserved preparatory to testing, as soon as they are cut from the sheet, in waxed or waterproofed string-tied envelopes to prevent any change in moisture content.

Moisture Content of Felt: With the least possible delay after the rolls have been sampled, cut from each square foot specimen a 2-in. strip (within $\frac{1}{2}$ in.) across the fiber grain. Weigh the strips in aggregate, and then heat for not less than one hour in an oven having a free circulation of air, the internal temperature of which is uniformly maintained between 215 and 225° F. Remove the felt from the oven and weigh quickly, to prevent reabsorption of moisture. Replace the specimens in the oven and reweigh at intervals of fifteen minutes, until no further loss is noted. Compute the average percentage by weight of moisture in the felt as received.

Another method³¹ consists in distilling a weighed quantity of the felt with perchloroethylene ($\text{CCl}_2.\text{CCl}_2$), collecting the distillate in a graduated tube and measuring the volume of water which floats on the surface.

Corrected "Number of Felt": The "corrected number" represents the weight of the moisture-free felt in pounds per 480 sq.-ft. The minimum, maximum and average "corrected number" of the felt is obtained by deducting the average percentage of moisture from the "uncorrected number."

(III) *For Textiles:* The "number" represents the weight in ounces per lineal yard of a specified width, the moisture content being arbitrarily fixed at 6.5 per cent. With burlaps the width is 40 in., with "regular" ducks 29 in., etc.

The following method of test has been standardized:³²

Unless otherwise specified, samples for moisture content are conditioned and the results based on the weight of the conditioned material.

When the moisture content of the material is specified, then representative samples of the material to be tested for moisture content shall be weighed at the time and under the conditions of sampling. They are submitted to the laboratory in sealed moisture-proof containers. The samples are acceptable for test only if the difference between the weight of the unopened container as received by the laboratory and the weight of the container after removal of the sample is within ± 0.2 per cent of the submitted weight of the sample. The results of the analysis are based on this weight.

A glass weighing bottle of approximately 100-ml. capacity fitted with a ground-glass cover is dried at 221 to 230° F. (105 to 110° C.) to constant weight. A satisfactory procedure is to place the bottle and cover separately in the oven. After drying for 1 hour they are transferred to a desiccator and allowed to cool to room temperature. The cover is placed on the bottle which is then weighed. The heating, cooling, and weighing are repeated until the weight is constant to ± 0.003 g. This is the "weight of weighing bottle." This weighing bottle is kept in a desiccator when not in use.

A specimen of approximately 5 g. of the material to be tested is placed in the weighing bottle which is then covered and weighed. This is the "weight of weighing bottle plus specimen." By subtracting the "weight of weighing bottle" from this weight, the "weight of specimen" is obtained.

The specimen is removed from the weighing bottle, placed on a watch glass in the oven (temperature 221 to 230° F. as before), dried for 1.5 hours, and quickly transferred to the weighing bottle, which is placed uncovered in a desiccator. When the specimen and bottle have cooled to room temperature, the cover is replaced on the bottle and it is weighed. The specimen is then returned to the oven and the drying, cooling, and weighing are repeated until the weight is constant to ± 0.003 g. This is the "weight of dry specimen and weighing bottle." By subtracting the "weight of weighing bottle" from this weight, the "weight of dry specimen" is obtained.

The moisture content of the specimen is given by the following calculation:

$$\left. \begin{array}{l} \text{Moisture content of} \\ \text{specimen in per cent} \end{array} \right\} = \frac{\text{Weight of specimen} - \text{Weight of dry specimen}}{\text{Weight of specimen}} \times 100$$

Test 78. Tensile Strength. The strength is determined by one of the following methods:

(1) *For Paper:* The sample after being "conditioned"³³ may be tested for tensile breaking strength by the following method which has been standardized:

(a) A testing machine of the dead-weight pendulum type suitably designed for testing paper shall be used. The machine shall preferably be power driven.

(b) The capacity of the machine shall not exceed 113 kg. (250 lb.).

(c) The machine shall be graduated to read 1 lb. or 1 kg. or less per scale division for testing paper breaking at 22.7 kg. (50 lb.) or over, and to 0.5 lb. or 0.5 kg. or less for testing paper breaking under 22.7 kg. (50 lb.).

From each of the samples selected, specimens shall be cut, at least ten in the machine direction and, if practicable, ten in the cross-machine direction. The specimens shall not exceed 2.54 cm. (1 in.) in width and shall be 25.4 cm. (10 in.) in length with clean-cut edges.

The ratio of the clearance distance between jaws to the width of the specimen shall not be less than 5 to 1, nor more than 10 to 1. The rate of travel of the movable jaw shall be constant. It shall preferably be 30.5 cm. (12 in.) per minute, but it may be within the limits of 28 cm. (11 in.) and 33 cm. (13 in.) per minute, provided it is constant.

All readings obtained when the paper breaks at or in the jaws shall be rejected. The results of the machine-direction specimens and the cross-machine-direction specimens shall be reported separately. The results shall be reported in kilograms or pounds together with the width of the specimen in centimeters or inches and also the average thickness. The maximum, minimum, and average

breaking load shall be reported for the machine direction and the cross-machine direction.

The bursting strength of the paper may be ascertained by the Mullen Tester (Fig. 365) or an analogous type of tester, the procedure having similarly been standardized as follows:

The testing machine shall have a circular flexible diaphragm 6.44 sq. cm. (1 sq. in.) in area. The pressure chamber shall be filled with glycerin or other suitable pressure medium and shall contain no air spaces. The test specimen shall be held in position over the diaphragm in a clamp having a circular hole approximately 6.44 sq. cm. (1 sq. in.) in area, so that the diaphragm will force the paper into the hole when pressure is applied under the diaphragm. The pressure exerted on the diaphragm shall be indicated on a tube-type of gage, graduated to at least 0.23 kg. (0.5 lb.) for papers giving bursting strengths 18 kg. (40 lb.) or under. A tube-type of gage graduated 0.45 kg. (1 lb.) may be used in testing papers which give higher bursting strengths. The machine may be either hand-operated or power-driven, the latter being preferable.

If practicable, the specimen shall be so cut from the stock as to permit ten bursting tests on a line across the sheet or roll. Ten bursts shall be made, five with one side of the paper uppermost and five with the other side uppermost. The testing machine shall be driven at a uniform speed of 120 r.p.m. until the specimen bursts. The report shall include the average, the maximum, and the minimum results obtained.

(II) *For Felt*: Expose the desaturated fabric for three days to air at 77° F., completely saturated with moisture, and then find its tensile strength as in Test 67. The reason for this is because the strength of the dry felt is increased materially during the process of extracting with solvents, but it may again be brought to correspond closely with its original strength by treating as described.

The following figures will illustrate this point, viz.: strength original felt (before extraction), 23.3 lb. (average of 10 tests); original felt upon heating to 265° F. for five minutes, cooling in a desiccator and testing immediately, 26.9 lb.; original felt upon extracting with benzol in Soxhlet for five hours, cooling in desiccator

and testing immediately, 36.4 lb.; extracted felt exposed three days to air at 77° F. carrying 30 per cent moisture, 33.6 lb.; extracted felt exposed three days to air at 77° F. completely saturated with moisture, 27.6 lb.

(III) *For Textiles* (e.g., cotton cloth, duck, burlap, etc.) : It is customary to find the tensile strength when the desiccated fabric has a moisture regain, which it assumes on exposure for at least four hours to an atmosphere having a relative humidity of 65 per cent at 70° F. (21.1° C.).

For the determination of strength and elongation of textile fabrics, the following methods of test have been standardized:³⁴

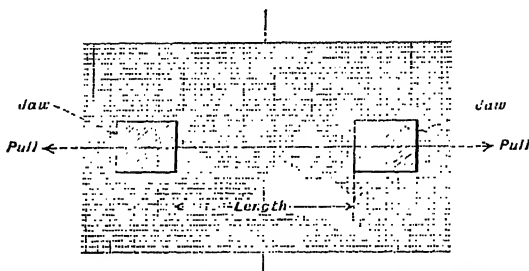
Breaking Strength (Grab Method) : Specimens 4 in. in width and not less than 6 in. in length shall be taken for test. Two sets of five specimens each are required, one set for warp breaking strength having the longer dimension parallel to the warp yarns and the other set for filling breaking strength, having the longer dimension parallel to the filling yarns. No two specimens for warp breaking strength shall contain the same warp yarns, or for filling breaking strength the same filling yarns. Unless otherwise specified, specimens shall be taken no nearer the selvage than one-tenth the width of the fabric.

A tensile testing machine conforming to the requirements of A.S.T.M. Designation : D 76 shall be used. The distance between the clamps at the start of the test shall be 3 in. The face of one jaw of each clamp shall measure 1 by 1 in., that of the other jaw of each clamp 1 by 2 in. or more, with the longer dimension perpendicular to the direction of application of the load.

The specimen shall be placed symmetrically in the clamps of the machine (see Fig. 374), with the longer dimension parallel to and the shorter dimension at right angles to the direction of application of the load, care being taken to grip the same yarns in both clamps. The average of the results of the five individual tests on the warp shall be reported as the warp breaking strength, and the average of the five individual tests on the filling shall be reported as the filling breaking strength. If a specimen slips in the clamps, breaks in the clamps, breaks at the edge of the clamps, or if for any reason attributable to faulty operation, the result falls

markedly below the average for the set of specimens, the result shall be discarded, another specimen taken, and the result of this break included in the average.

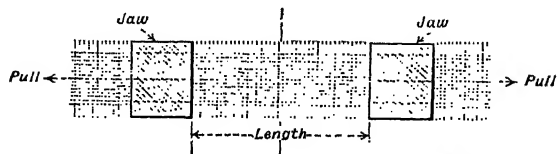
Raveled-strip Method: Shall be made in accordance with the directions for the grab method with the following exceptions: The specimens shall be $1\frac{1}{4}$ in. in width if there are 50 or more yarns



Courtesy A.S.T.M.

FIG. 374.—Illustration of Grab Test.

per inch, and $1\frac{1}{2}$ in. in width if there are less than 50 yarns per inch. Each specimen shall be raveled to 1 in. in width by taking from each side approximately the same number of yarns. The clamps used on the testing machine shall have faces measuring 1 by $1\frac{1}{2}$ in. or more, the longer dimension being perpendicular to the direction of application of the load (see Fig. 375).



Courtesy A.S.T.M.

FIG. 375.—Illustration of Strip Test.

Cut-strip Method: Shall be made in accordance with the directions for the raveled-strip method with the exception that the specimens shall be cut 1 in. in width (or other width when specified). This method is applicable to heavily sized or coated fabrics which cannot be tested by the raveled-strip method.

Elongation of Fabrics: Unless otherwise specified, the elonga-

tion of fabric at any stated load shall be obtained when the breaking strength is determined and for the same specimens by means of a suitable autographic recording device on the testing machine. The elongation shall be the average of the results obtained for five specimens, and it shall be expressed as the percentage increase in length. Since the initial length and, therefore, the measured elongation depend upon the load applied in placing the specimen in the clamps of the machine, an initial load of 6 oz., or other initial load specified for the particular material in question, shall be placed on the specimen before gripping the specimen in the lower clamp of the machine. The elongation shall be calculated from the start of the line as shown on the graphic record.

Test 79. Tearing Resistance of Textiles or Paper. The tearing resistance is determined by the following methods:

(1) *For Textiles:* The test specimens shall be 3 in. in width and 6 in. in length. Two sets of five specimens each are required, one set for warp tearing strength, having the longer dimension parallel to the warp yarns, and the other set for filling tearing strength, having the longer dimension parallel to the filling yarns. An isosceles trapezoid having an altitude of 3 in. and bases 1 and 4 in. in length shall be marked on each specimen, preferably with the aid of a template. A cut $\frac{1}{4}$ to $\frac{3}{8}$ in. in length shall then be made in the center of the 1-in. edge and perpendicular to it.

The testing machine used for this test shall conform to the requirements of the machine used in the grab method for breaking strength with the following modifications: The faces of the clamps shall measure 1 by 3 in. or more, with the longer dimension perpendicular to the direction of application of the load. The distance between the clamps at the start of the test shall be 1 in. If the machine is of the pendulum type, the pawls on the pendulum shall be disengaged from the ratchet.

The specimen shall be clamped in the machine along the non-parallel sides of the trapezoid so that the cut is halfway between the clamps, the short edge shall be held taut, the long edge lying in folds. The machine shall be started and the average load necessary to tear the fabric shall be observed, preferably by means of an autographic recording device. The average of the results of the five individual tests on the warp shall be reported as the warp

tearing strength, and the average of the five individual tests on the filling shall be reported as the filling tearing strength.

Various other testing procedures adapted to particular textile fabrics have similarly been standardized.³⁵

(II) *For Paper:* The following procedure has been standardized:³⁶

This method covers the procedure for determining the average force in grams required to tear a specimen of paper.

The testing machine shall be of the pendulum impulse type and shall consist of the following:

(a) A stationary clamp,

(b) A movable clamp carried on a pendulum, preferably formed by a sector of a wheel or circle, free to swing on a ball bearing or other substantially frictionless bearing,

(c) Means for holding the pendulum in a raised position and means for releasing it instantaneously, and

(d) Means for registering the maximum arc through which the pendulum swings when so released. The pendulum shall carry a circumferential scale, graduated from 0 to 100, so as to read against the pointer the work done in gram-centimeters by the pendulum when tearing a specimen of paper, divided by 137.6 (one-sixteenth of the force in grams required to tear a specimen).

With the pendulum in its initial position ready for a test, the two clamps shall be separated by an interval of 2.5 mm. (0.10 in.) and so aligned that the specimen clamped in them lies in a plane perpendicular to the plane of oscillation of the pendulum with the edges of the jaws gripping the paper in a horizontal line, a perpendicular to which through the axis of suspension of the pendulum is 104 mm. (4 in.) in length and makes an angle of 27.5 deg. with the plane of the paper.

The clamping surface in each jaw shall be over 25 mm. (1 in.) in width and over 12 mm. (0.5 in.) in depth.

For apparatus having a sector as a pendulum, and which tears the specimen as it moves toward the right, draw a pencil line on the base or stop-mechanism 1 in. to the right of the edge of the sector stop. With the sector raised to its initial position and the pointer set against its stop, on releasing the sector and holding the stop down, the sector should make at least 20 complete oscillations before the edge of the sector which engages with the stop no longer

passes to the left of the pencil line. Otherwise, the bearing shall be oiled and adjusted.

Level the instrument so that, with the sector free, the line on the sector indicating the vertical from the point of suspension coincides with a corresponding point on the base of the instrument, usually placed on the stop mechanism. After leveling, operate the instrument several times with nothing in the jaws, the movable jaw being closed, to ascertain if the pointer registers zero with no load. If zero is not registered, the pointer stop should be suitably adjusted until the zero reading is obtained. If it is necessary to move the pointer stop, the pointer friction should be checked as follows: Set the pointer at the zero reading on the scale before releasing the sector and after release see that the pointer is not pushed more than three scale divisions beyond the zero. A reading of more than three divisions indicates excessive pointer friction and the pointer should be removed, the bearing wiped clean, and a trace of oil or petroleum jelly applied. When the pointer friction has been reduced, finally adjust the pointer stop.

Level and adjust the instrument on a level sheet of plate glass and clamp a known weight in grams, W , to the radial edge of the sector beneath the jaws. The center of gravity of the weight (including means of attaching) shall be previously marked by a punched dot on the face of the weight that is to be in the front of the instrument.

Raise and set the sector as for tearing a sheet and, by means of a surface gage or other convenient means, measure the height in centimeters, H , of the center of gravity of the weight above the glass plate. Then release the sector, allow it to swing, and note the pointer reading. Without touching the pointer, raise the sector until the edge of the pointer just meets with its stop, in which position again determine the height in centimeters, h , of the center of gravity of the weight above the glass plate.

The work done is $W(H - h)$ gram-centimeters. The pointer reading for the standard instrument and method specified should be as follows:

$$\frac{W(H - h)}{137.6}$$

Five weights from 75 to 400 g. form a suitable range for the calibration, one or more being clamped on the edge of the sector in different positions, the work done in raising each being calculated and added together.

A record shall be made of deviations of the indicated readings and corresponding corrections made in the test results.

It is unnecessary to repeat the calibration of the instrument provided it is kept in adjustment and no parts become changed or worn, but the tearing distance, which equals 43 mm. (1.69 in.), shall be checked prior to each series of tests and adjusted, if necessary.

Test specimens shall be cut accurately in each principal direction of the paper about 76 mm. (3 in.) in length by exactly 63 mm. (2.5 in.) in width with the slit to be cut 20 mm. (0.8 in.) in length, leaving exactly 43 mm. (1.69 in.) between the end of the slit and the edge of the specimen.

Level and adjust the testing machine, if necessary, before each set of tests.

Place the test specimen midway in the clamps with its upper edge parallel to the top of the jaws and so that the initial slit is at right angles to the top of the jaws. Make alternate tests with the wire sides of the sheets comprising the test specimen facing opposite directions. Test enough sheets so that, when torn together, the scale readings are between 20 to 60. Record the number of sheets so tested (1 to 16 sheets may be used). Make not less than five tests in each principal direction of the paper.

If the mean value of the lowest and the highest reading differs from the average of all the readings by more than 10 per cent, test additional specimens until there is agreement within these limits. Discard an isolated very high or low result that is not repeated in duplicate when a consistent average has been obtained without the abnormal reading. Reject readings obtained where the tear deviates more than 10 mm. ($\frac{3}{8}$ in.) from the line of the initial slit. If results deviate more than this, a notation should be made and the deviation reported. If the side of the specimen above the movable clamp rubs against the sector as a tear is made, reject that reading.

Report the results as the force in grams required to tear a speci-

men. Since the scale readings are made one sixteenth of the actual values, the tearing resistance values shall be calculated by multiplying the average instrument reading (corrected if necessary for calibration error) by 16 and dividing by the number of sheets torn at one time. Report the results as follows:

Result	Report to Nearest
Below 10 units.....	0.1 g.
10 to 19.9.....	0.2 g.
20 to 49.9.....	0.5 g.
50 and above.....	1 g.

Report the average, maximum, and minimum results for both principal directions of the paper and also the number of sheets torn at one time. Report results obtained on strips torn in the machine direction as resistance to tearing in the machine direction, and report results obtained on strips torn across the machine direction as resistance to tearing in the cross direction.

Duplicate determinations on different sets of specimens from the same shipment and on different testing instruments should agree with each other within 7 per cent.

Test 79a. Folding Endurance of Paper. The following procedure has been standardized:³⁷

These methods of test for measuring the folding endurance of paper cover two test procedures, as follows:

Method I: Schopper folding endurance.

Method II: M.I.T. (Massachusetts Institute of Technology) folding endurance.

The Schopper apparatus is applicable for testing papers having a thickness of not over 0.01 in. The M.I.T. apparatus can be adjusted for testing papers of any thickness. There is no constant relation between the values obtained with the two types of apparatus.

Method (I). Schopper Folding Endurance: The apparatus shall consist of the following:

(a) Schopper Tester: Two horizontally opposed clamps, approximately 10 cm. apart, provided with spring tension which varies during the folding cycle as a slotted folding blade, sliding back and forth between creasing rollers, folds the paper. The clamps, while in motion, shall be freely suspended between the tension springs, except that they shall be supported from below

by rollers. The folding blade shall be 0.50 mm. (0.02 in.) in thickness, and the edges of the vertical folding slot shall be cylindrical and shall extend somewhat above and below the normal position of the test specimen. The four creasing rollers, each approximately 6 mm. (0.24 in.) in diameter and 18 mm. (0.71 in.) in length, shall be arranged symmetrically about the mid-position of the folding slot, and shall preferably be provided with jewel bearings.

(b) Motor: A means of imparting harmonic motion of constant period to the reciprocating blade. A power-driven apparatus is preferable.

(c) Counter: A device for registering the number of double folds, which stops automatically when the specimen is severed.

Test the clamps by fastening a specimen in place in the manner described, alternately applying and releasing the tension a number of times. Then, with tension released, note whether the specimen remains smooth and straight as originally inserted. Buckling or waviness indicates a faulty clamp which will allow the specimen to slip.

Inspect all rollers for worn surfaces and for bearing friction, and make the necessary corrections. Adjust the supporting rollers so that they do not bind against the clamps in any position. With leaf gages inspect the four creasing rollers for parallelism and clearances. Also, make sure that the two edges of the folding slot are parallel with each other and with the creasing rollers. Adjust the distance between the folding blade and the two creasing rollers on each side to 0.38 mm. (0.015 in.), and the width between rollers of the space occupied by the unbent specimen to approximately 0.5 mm. (0.02 in.). As a final test of alignment, fold a specimen somewhat short of failure and inspect of it for uniformity of wear along the crease. If the specimen seems weaker at one end of the crease than at the other, faulty alignment of the rollers or the folding slot is indicated (if the clamps have been properly adjusted), which may result in low values for folding endurance.

The roller friction may be measured by means of the bell-crank-lever weighing device, as follows: First, set a pair of bow dividers to show the displacement of each clamp when loaded directly with 1 kg. Then, shift the weighing device 90 deg. so as to load a clamp through a ribbon passed around one of the creasing rollers. Add

weights in excess of 1 kg. until the direct 1-kg. displacement is reproduced. This excess weight is a measure of the roller friction in terms of the increased tension it will produce. Repeat the measurement for the other three rollers. The excess weight required shall not be greater than 100 g.

Adjust the tension spring attached to the clamps against a dead-weight load so that the tension on the specimen during a test is 790 g. when the clamps are farthest apart (and when the specimen is straight and free) and $1 \text{ kg.} \pm 50 \text{ g.}$ when they are nearest together. Make adjustments, preferably on the assembled instrument with the aid of a suitable weighing device, such as a balanced bell-crank lever with knife-edge fulcrums at the center of gravity, capable of balancing the tension of a horizontal spring against the weight of a known mass. Fasten a strip of strong paper or celluloid, about 0.005 in. (0.127 mm.) in thickness in the clamps and apply the tension. Set a pair of bow dividers (by spanning the distance between two suitably placed fiducial marks, such as small punch marks) to show the displacement of each clamp. With a load of 790 g. acting on one clamp and spring, adjust the spring until this displacement is reproduced. Repeat for the other spring. To verify the tension at maximum displacement, set a pair of dividers to show the displacement of each clamp when the folding blade has pushed the crease in the specimen to the end of its stroke each side of the midposition (four measurements). With the aid of the weighing device, load each clamp until this displacement is reproduced in each case. The load required in each case should be approximately 1 kg.

Apparatus in steady use shall be adjusted and calibrated at intervals of not more than 1 month.

Test specimens $15 \pm 0.25 \text{ mm.}$ ($0.59 \pm 0.01 \text{ in.}$) in width and 10 cm. (4 in.) in length shall be cut accurately from the sample in each principal direction of the paper. The specimens shall be initially free from folds, wrinkles, or blemishes not inherent in the paper, and the area in which the flexing is to take place shall not contain any portion of the watermark. The edges of the specimens shall be clean-cut and parallel to the opposite edge. At least 10 specimens cut from each principal direction of the paper shall be tested.

All test specimens shall be brought to a standard condition, prior to testing, in accordance with A.S.T.M. Designation: D 685.

With the vertical slot of the reciprocating blade in its central position, place the specimen in the slot and fasten the ends firmly and squarely in the clamps with the surface of the specimen lying wholly within one plane. Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the prescribed tension and fold the specimen at a uniform rate of approximately 120 double folds per minute until it is severed at the crease. Record the number of double folds required to sever the specimen.

The number of double folds required to sever the specimen shall be reported as Schopper folding endurance (double folds) and shall include the number of tests, and the average, maximum, and minimum number of folds for each of the principal directions of the paper. Specimens tested cut with their length in the machine direction of the paper shall be designated as "machine direction." Specimens tested with their length cut at right angles to the machine direction of the paper shall be designated as "cross machine direction." In reporting average results, all digits after the first two shall be rounded off to zero.

Method (II). M.I.T. Folding Endurance: The apparatus shall consist of the following:

(a) M.I.T. Tester: A loading clamp constrained to move without rotation in a direction perpendicular to the axis of rotation of the folding head and having its clamping surfaces in the plane of this axis. The load shall be applied through a spring attached to the loading clamp which shall be easily adjustable to provide any desired tension on the specimen from 0 to 1.5 kg. The deflection of the spring when loaded shall not be less than 17 mm. (0.67 in.) per kg.

An oscillating folding head which shall support two smooth, cylindrical folding surfaces parallel to, and symmetrically placed with respect to, the axis of rotation. The position of the axis of rotation shall be approximately in the common tangent plane to the two folding surfaces in the conventional design, and midway between them. The folding head shall be provided with a clamping device back of the axis of rotation and so designed that no clamping pressure is exerted nearer than $\frac{3}{8}$ in. (3.18 mm.) to the bend-

ing axis. The rotary oscillating movement of the folding clamp shall be such as to fold the paper through an angle of 135 ± 5 deg. to both right and left of the position of zero fold. Each of the two folding surfaces shall have radius of curvature of 0.38 ± 0.015 mm. (0.015 ± 0.001 in.) and a length of not less than 19 mm. (0.75 in.). The distance separating the folding surfaces shall be greater than the uncompressed thickness of the paper being tested, but shall not exceed it by more than 0.25 mm. (0.01 in.).

(b) Motor: A motor-driven device for imparting a rotary oscillating motion of constant period to the folding clamp.

(c) Counter: A device for registering the number of double folds required to sever the specimen.

All working parts of the apparatus shall be in good condition, well oiled, and in proper adjustment. Particular care shall be given to make certain that the folding edges are free from rust or dirt.

Measure the plunger friction by determining the additional load required to move the plunger perceptibly when displaced under a load of 1.0 kg. or the load tension used in testing. The additional load required shall not be greater than 25 g.

Measure the change in tension due to eccentricity of rotation of folding edges as follows: Place a strip of strong paper, cut in the machine direction and of the proper thickness, in the tester in the same manner in which a folding test would be made, and apply a tension of 1.0 kg. or that prescribed for the test. Rotate the folding head slowly throughout the entire folding cycle and measure the maximum change in displacement of the plunger to an accuracy of 0.1 mm. (0.004 in.). The amount of load required to produce the same displacement shall not be greater than 35 g. Measure the curvature of the folding edges by comparing suitable casts magnified in profile to standard-circles.

Apparatus in steady use shall be adjusted and calibrated at intervals of not more than 1 month.

Test specimens 15 ± 0.25 mm. (0.59 ± 0.01 in.) in width and at least 14 cm. (5.5 in.) in length shall be cut accurately from the sample in each principal direction of the paper. The specimens shall be initially free from folds, wrinkles, or blemishes, and the area in which the flexing is to take place shall not contain any portion of the watermark. The edges of the specimens shall be clean-

cut and parallel to the opposite edge. At least 10 specimens cut from each principal direction of the paper shall be tested.

All test specimens shall be brought to a standard condition prior to testing and all tests shall be made under standard atmospheric conditions.

Place the oscillating folding head in the position of zero fold. Place on top of the plunger a weight equivalent to the tension desired on the specimen and clamp the plunger in position when depressed under this load. Then clamp the specimen firmly and squarely in the jaws with the surface of the specimen lying wholly within one plane and not touching the jaw mounting-plate. Handle the specimen by the ends and do not touch it with the hands in the region which is to be folded. Then apply the prescribed tension to the specimen by releasing the plunger. If the reading of the load indicator has changed, reset it by means of the adjusting screw to agree with the reading obtained when the weight was applied. Whenever possible, a tension of 1.0 kg. shall be used, but if this does not afford practical test results, a greater or a lesser tension may be used. Fold the strip at a uniform rate of 175 double folds 15 per cent per minute until it is severed at the crease. Record the number of double folds required to sever the specimen.

The number of double folds required to sever the specimen shall be reported as the M.I.T. Folding Endurance (double folds) *at the tension used*, and shall include the number of tests, and the average, maximum, and minimum number of folds for each for the principal directions of the paper. Specimens tested with their length cut in the machine direction shall be designated as "machine direction." Specimens tested with their length cut at right angles to the machine direction shall be designated as "cross machine direction." In reporting average results, all digits after the first two shall be rounded off to zero.

Test 80. Porosity of Paper or Felt. This test measures the porosity as indicated by the "air resistance," by an instrument known as the "densometer," illustrated in Fig. 376. The procedure has been standardized as follows:⁸⁸

The apparatus shall consist of two aluminum open-top cylinders, one of which is inverted and slides into the other which is fixed. The movable cylinder shall be provided with a circular aperture

in the closed end and a flat ring clamp for holding the specimen across this aperture.

The fixed cylinder shall be 25.4 cm. (10 in.) in height and shall have an external diameter of 8.60 cm. (3.38 in.) and an internal diameter of 8.25 cm. (3.25 in.). Four slender bars, each 18.5 cm. (7.3 in.) long, 3 mm. (0.12 in.) wide and approximately 1.5 mm. (0.06 in.) thick shall be mounted vertically and equidistantly on the inner surface of the fixed cylinder to act as guide-tracks for the movable cylinder. The movable cylinder shall be graduated in units of 50 ml. and shall have a total range of 350 ml. It shall be 25.4 cm. (10 in.) high and shall have an external diameter of 7.62 cm. (3.00 in.) and an internal diameter of 7.35 cm. (2.90 in.). It shall weigh, including the flat ring clamp and the two knurled nuts, 567 g. \pm 0.5 g. (20 oz. \pm 0.018 oz.). The movable cylinder and the flat ring clamp shall have a concentric circular aperture of 6.44 sq. cm. (1.00 sq. in.) in area. When this aperture is too large for the specimen, a similar movable cylinder and flat ring clamp having a concentric circular aperture of 1.61 sq. cm. (0.25 sq. in.) in area shall be used.

NOTE.—Results obtained with apertures of different areas are not directly proportional to the areas of the apertures. The proportionality factor must be determined by experiment.

The specimens shall be not less than 3.5 cm. (1.36 in.) nor more than 5.1 cm. (2.0 in.) in width cut from the original samples, and shall be as long as the width of the original roll of paper. If the specimens are selected from pads of tape they shall be not less than 2.1 cm. (0.81 in.) nor more than 5.1 cm. (2.0 in.) wide and at least 30 cm. (12 in.) long. There shall be as many specimens as there are original samples.

The fixed cylinder shall be placed on a rigid support so that its sides are vertical. A lubricating oil with viscosity of 60 to 70 sec-



Courtesy W. & L. E. Gurley

FIG. 376.—The Densometer.

onds Saybolt at 37.8° C. (100° F.) shall be placed in it, to a depth of 12.7 cm. (5 in.). The specimen (one thickness only) shall be secured tightly under the clamp on the movable cylinder completely covering the aperture. The movable cylinder is then floated on the lubricating oil. The time required for the displacement of a certain amount of air shall be noted with a stop-watch. If possible, the amount of air displaced should be such that the time of displacement is not less than 20 seconds. The apparatus with its content of lubricating oil shall be at the temperature of the conditioning room when the readings are taken. The time in seconds required for the displacement of 100 ml. (6.1 cu. in.) of air through a circular area (one side only) of 6.44 sq. cm. (1 sq. in.) of the paper is known as the air resistance of the paper.

NOTE.—The clamp shall be tested for leakage by substituting a piece of tinfoil 0.05 mm. (0.002 in.) in thickness for the paper and testing in the manner described above. When so tested, the leakage shall not exceed the rate of 50 ml. in five hours.

The proper procedure for clamping the specimen or tinfoil is to turn both knurled nuts down onto the clamp simultaneously. If only one nut at a time is turned down, the clamp will not fit flat on the specimen and will consequently have an avoidable leak.

Oil is used in preference to distilled water because it does not corrode aluminum.

Precautions should be taken to avoid subjecting the apparatus to vibration as this condition would increase the rate of air displacement.

The report shall include the following:

- (a) the number of seconds required for the displacement of 100 ml. (6.1 cu. in.) of air;
- (b) the area of paper through which the air was displaced;
- (c) the thickness of the paper;
- (d) the room temperature.

NOTE.—The following values show the probable accuracy obtainable in the air-resistance tests:

Air Resistance	Accuracy
40 seconds.....	± 5 per cent
100 seconds.....	± 6 per cent
200 seconds.....	± 8 per cent
300 seconds.....	± 10 per cent

Test 81. Saturating Speed of Felt. This test is designed to ascertain the relative speeds with which roofing felt will saturate with bituminous substances at operating temperatures. Two alternate procedures are recommended for the purpose:

(I) *Vertical Method*:³⁹ Strips of moisture-free felt 2.5 cm. wide cut lengthwise from the sheet (the length being immaterial) are marked with a pencil 1 cm. and 4 cm. from one end. They are then supported vertically and immersed up to the 1-cm. mark in xylol distilling between 135 and 145° C. by the standard Engler distillation method, which is maintained at 77° F. By means of a stop-watch the time is recorded during which the xylol rises by capillarity to the 4-cm. mark (i.e., exactly 3 cm. above the level of the xylol). The number of seconds is an index of the speed with which the felt will saturate.

(II) *Horizontal Method*:⁴⁰ Samples measuring 2 by 2 in. are gently floated on the surface of a mixture of clear paraffin oil with a clear light engine oil, having a Saybolt viscosity of 253 seconds at 72° F. and 125 seconds at 100° F. One specimen is floated right side up, and another upside down on the oil maintained at 72° F. When the uppermost surface is judged to be darkened by the oil to an extent of 90 per cent of its area, the time in seconds is recorded. The number of seconds for the oil to penetrate from each side of the sheet is termed the "oil penetration" for the respective side of the felt. To facilitate making the test, it is recommended that a series of oil mixtures be prepared, each having the prescribed Saybolt viscosity at 60°, 65°, 70°, etc., up to 85° F. respectively, so that the particular oil may be used, depending upon the room temperature, thus avoiding the necessity of bringing the oil to the proper temperature. The moisture content of the felt affects the results. It has also been suggested that the weight of oil absorbed be ascertained and expressed in per cent by weight of the dry felt.

Test 82. Saturation Capacity of Felt. This test is intended to serve as an indication of the quantity of saturant which the felt may be expected to absorb under definite machine conditions. It consists in saturating a sample of the moisture-free felt with a measured volume of kerosine, and hence has been termed the "kerosine test,"⁴¹ which has been standardized as follows:

This method of test for the absorptive qualities of felt, expressed as kerosine number, is applicable to felts used in the manufacture of roofing and flooring.

NOTE.—The kerosine number of a felt, as determined by this method, is based on the relation between the specific gravity of the kerosine used and the specific gravity

of water (1.00). To obtain the relation between the saturating capacity of the felt and any bituminous saturant to be used with the felt, the specific gravity at 77° F. (25° C.) of the bitumen shall be determined in accordance with the Standard Method of Test A.S.T.M. Designation: D 70 and that figure multiplied by the "kerosine number" of the felt, and expressed as a percentage figure.

The kerosine number of roofing and flooring felt is calculated from the maximum weight of a kerosine oil, of known specific gravity, retained by the felt after displacement of all the air from the interior voids. It is a measure of the amount of saturant which a given felt will absorb.

The apparatus shall consist of the following:

- (a) Balance: Analytical balance, sensitive to 1 mg.
- (b) Oven: Laboratory oven, steam or electrically heated and capable of maintaining a temperature between 220 and 225° F., inside dimensions to be not less than 12 by 12 by 12 in.
- (c) Weighing Container: Lightweight glass or metal container with tight-fitting cover for weighing the specimens, and of a suitable size to hold the specimens in a horizontal position without bending or distorting them.
- (d) Thermo-hydrometer: Glass hydrometer and thermometer combined, also a glass hydrometer cylinder as prescribed in A.S.T.M. Designation: D 287.
- (e) Vacuum Pump: A vacuum pump capable of reaching and maintaining a vacuum of not less than 28 in. of mercury in the test cylinder.
- (f) Vacuum Cylinder: A glass vessel of approximately 1500 cu. cm. capacity for soaking the felt specimens in kerosine under vacuum.
- (g) Desiccator: A desiccator of size suitable for cooling felt strips in the weighing containers.

The water-white kerosine used in the test shall have a specific gravity between 0.776 and 0.825 at 25° C. (77° F.).

Six representative specimens, each measuring $2 \pm \frac{1}{8}$ in. by $5 \pm \frac{1}{8}$ in. in size, shall be selected from the felt to be tested. The specimens shall be cut with the 5-in. side running parallel to the machine direction of the sheet.

Place the test specimens in the tared weighing container and expose them, uncovered, for not less than 1 nor more than 2 hrs. in the oven, the interior of which is maintained uniformly at a temperature between 220 and 225° F. The specimens shall be kept

flat; they shall not be folded, rolled, or in any way distorted. Handle the six specimens as a unit in all of the following operations.

Remove the specimens from the oven in the weighing container and place, still uncovered, in the desiccator to cool.

Rapidly seal the weighing container with its cover and weigh the container and included specimens on the analytical balance to the nearest 10 mg. Calculate the net weight of the dry specimens.

Insert a wire hook in one corner of the specimens and immerse them at once in a vertical position in 1 liter of kerosine at $25 \pm 1^\circ$ C. ($77 \pm 1.8^\circ$ F.) in the glass vacuum vessel. Apply a vacuum of not less than 28 in. of mercury to the vessel containing the specimens and hold them under that condition for 15 min., or until the bubbles cease to come from the specimens, whichever period is the longer. Remove the specimens from the kerosine and permit them to drain in the machine direction of the felt fibers for 3 min. \pm 1 sec., allowing the lower corner of each specimen to touch the edge of the kerosine container.

Return the specimens to their weighing container, seal with its cover, and determine and record the combined weight of the specimens and the kerosine which they have absorbed.

Determine the specific gravity at 25° C. (77° F.) of the kerosine used in the test in accordance with A.S.T.M. Designation: D 287.

Calculate the kerosine number of the specimens as follows:

$$\text{Kerosine number} = \left(\frac{b}{a} - 1 \right) \times \left(\frac{1}{g} \times 100 \right)$$

where a = weight of dry felt,

b = weight of felt plus absorbed kerosine, and

g = specific gravity of kerosine at 25° C. (77° F.)

Report the results of the kerosine number determinations to three significant figures.

Test 83. Fiber Composition. The following methods have been proposed.⁴² The equipment shall consist of the following:

Microscope: A microscope capable of giving not less than 100 diameters magnification is necessary for the determination of fiber composition. It is desirable that the microscope be of the compound type and that it be equipped with a mechanical stage.

Cross-line Disc: This disc is employed to establish a dot or point for counting the fibers passing under it.

Dropper: This shall consist of a glass tube 6 in. (20 cm.) long and $\frac{1}{4}$ in. (6 mm.) internal diameter, fitted at one end with a rubber bulb and having the other end carefully smoothed but not constricted.

The prepared slides shall be examined microscopically, using a magnification of not less than 100 diameters. Place the stained slide on the mechanical stage of the microscope, and count the fibers at various points in a straight line, twice lengthwise and four times crosswise of the slide, starting each line of observation at a different point. The number of each kind of fiber present in at least 25 different fields and a total of not less than 600 fibers, 200 on each slide, shall be counted.

Place a cross-line disc on the diaphragm of the eyepiece of the microscope. As each fiber passes under the dot or point formed by the intersecting lines on the disc, count it as one, regardless of its size. If aggregations of fibers such as occur in ground wood are encountered, the number of single fibers in the aggregation shall be estimated and counted as if the fibers were completely separated.

The proportion of the various fibers found shall be reported in terms of percentages of the total fiber composition, to the nearest 5 per cent.

A piece about 5 mm. square shall be cut from each of 10 sheets of the delivery sample being tested. These pieces shall be placed in a 50 or 100 cc. beaker or Erlenmeyer flask with approximately 20 cc. of 2 per cent solution of potassium hydroxide, then boiled, and washed thoroughly with water. This sample shall then be rolled into a ball and worked between the fingers to loosen the fibers. This can best be done by rolling between the index finger and thumb. The ball of paper shall then be placed in a test tube approximately 15 by 125 mm. Fill tube about three-fourths full with water and shake thoroughly until fibers are completely separated. After shaking, transfer about 5 cc. of the thoroughly mixed pulp to another test tube and fill tube about three-fourths full with water and shake well. As small a sample as can be conveniently handled shall be removed with needles or fine forceps, placed

on a glass slide, and water removed by means of hard filter or blotting paper.

The slide shall be prepared by removing several samples of pulp from test tube as described under preparation of sample, and stained in accordance with details of the respective methods outlined under stains. The prepared slide shall be examined by means of the microscope, the slide being moved systematically so that the whole slide is covered. The percentage fiber determination shall be made only by thoroughly trained analysts familiar with the fiber analysis of paper. The determination of the percentage of pulp shall be made by a recognized standard method and at least 300 fibers counted. Report the determined percentage of each kind of fiber on the basis of 100 per cent fiber.

(I) *General Stain (Herzberg Stain)*:⁴³ This stain is used for differentiating between all kinds of fibers as stated below. The composition of this stain is as follows:

(a) An aqueous solution of C.P. zinc chloride saturated at 70° F.

(b) 0.25 g. of C.P. iodine and 5.25 g. C.P. potassium iodide dissolved in 12.5 cc. of distilled water.

Mix 25 cc. of solution (a), measured at 70° F., with solution (b). Pour into a narrow cylinder and allow to stand until clear. Decant the supernatant liquid into an amber-colored, glass-stoppered bottle and add a small piece of iodine to the solution. Thoroughly moisten the fibers with this solution and remove the excess with blotting paper. The solution should be tested with known fibers and readjusted if necessary by addition of either zinc chloride or iodine. The following colors are developed by this stain:

Red—linen, cotton, bleached manila hemp.

Blue—chemically prepared fibers low in lignocellulose, from wood, straw, and esparto.

Yellow—fibers high in lignocellulose such as ground wood, jute, and unbleached manila hemp.

(II) *Distinguishing Between Coniferous and Deciduous Wood Fibers*:⁴⁴ The solutions required are:

- (a) 100 g. calcium nitrate in 25 cc. water.
- (b) Chlorzinc iodide solution (Herzberg's solution may be used).

The fibers are floated in 3 drops of the calcium nitrate solution for 1 min. Then 1 drop of chlorzinc iodide solution is added. After a few minutes the characteristic colors are established.

The fibers of coniferous wood turn pink, whereas the fibers of deciduous wood turn blue.

(III) *Distinguishing Between Bleached and Unbleached Wood Fibers (Bright Stain)*:⁴⁵ This stain shall be used for differentiating between bleached and unbleached fibers. The solutions required are:

(a) 2.7 g. ferric chloride ($\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$) per 100 cc. distilled water.

(b) 3.29 g. potassium ferricyanide ($\text{K}_3\text{Fe}(\text{CN})_6$) per 100 cc. distilled water.

(c) 3 g. of crude (not treated with sodium carbonate) substantive red dye per 500 cc. of distilled water. The dye used shall be Benzo purpurine 4B concentrated. These solutions must all be made with cold water.

Filter solutions (a) and (b) and keep in separate stock bottles at temperature not exceeding 20° C. Make solution (c) fresh each day it is used. For staining, use tall narrow beakers, suspending the microscopic slides in the beakers from clamps. Mix 10 cc. each of solutions (a) and (b) in one beaker and add an equivalent amount of solution (c) in another beaker. Set the beakers in a water bath, the temperature of which must be maintained constantly within plus or minus 1° of 20° C. Place a thermometer in the stains. When their temperature is 20° C. dip the slide containing the dry fibers in distilled water to moisten it uniformly (so that no air bubbles will be formed when it is stained), then place the slide in stain (a-b) and allow it to remain 20 min. Wash by dipping in distilled water six times. Then renew the water and repeat the washing process. Dry the contents of the slide and repeat the process of moistening, washing, and drying, using the (c) stain.

The colors developed by the Bright stain are:

Red—bleached fibers or fibers practically free from lignocellulose.

Blue—unbleached fibers or fibers containing lignocellulose.

(IV) *Distinguishing Between Unbleached Sulfate (Kraft) and Unbleached Sulfite Wood Fibers (Lofton-Merritt Stain)*:⁴⁶ The stain shall be prepared as follows:

- (a) Malachite green, 2 g.; distilled water, 100 cc.
- (b) Basic fuchsin, 1 g.; distilled water, 100 cc.

These shall be mixed in the proportion of 1 part (a) to 2 parts (b). As dyes from different sources vary, it is necessary to test them by staining known fibers. Unbleached sulfate fibers are stained blue or blue-green and unbleached sulfite fibers purple or lavender. If any purple fibers appear in unbleached sulfate fibers, this indicates there is too much fuchsin present, and more malachite green solution must be added. The opposite is indicated if some unbleached sulfite fibers develop a green or blue color.

The Lofton-Merritt stain shall be used as follows: Add the compound stain to the fibers and allow to remain 2 min. Remove excess stain by means of a hard blotting paper and add a few drops of 0.1 per cent hydrochloric acid. After about 30 sec. remove excess of acid. Next, add a few drops of distilled water and remove the excess.

Unbleached sulfate fibers are stained blue or blue green and unbleached sulfite fibers purple or lavender.

(V) *Distinguishing Between Bleached Sulfate and Bleached Sulfite Wood Fibers*:⁴⁷ To distinguish between bleached sulfite and bleached sulfate (i.e., kraft) fibers, the following method has been proposed: One gram of sodium carbonate is dissolved in 175 ml. of distilled water, and to this solution 1 g. of C.P. brazilin* is added, stirring until dissolved. The solution gives sharper differences when used fresh, but if retained for future use, air should be excluded. The solution may be applied directly to the sample to be identified, the excess being removed with hardened filter paper, a few drops of U.S.P. white paraffin oil being placed on the slide, and any excess oil being removed.

* MacAndrews & Forbes, New York City.

Bleached sulfite is stained a wine-red color, and bleached sulfate is stained a purple color by the brazilin solution.

(VI) *Distinguishing Between Alpha, Beta and Gamma Cellulose Fibers*: A method of test has been standardized for this determination.⁴⁸

(VII) *Determining Pentosans in Paper*: A method has likewise been standardized for this test.⁴⁹

(VIII) *Determining Jute*: The following procedure has been proposed:⁵⁰ A stain is prepared composed of:

- (a) 1 g. of phloroglucinol dissolved in 80 ml. of alcohol.
- (b) Concentrated hydrochloric acid.

The fibers are placed in a drop of solution (a). Then a drop of solution (b) is added.

Unbleached jute fibers turn dark violet-red, because they are heavily lignified.

(IX) *Determining Flax and Hemp*: The following method has been proposed:⁵¹

A saturated solution of cyanin (Colour Index No. 806) is prepared at ordinary temperature. This is diluted somewhat with water. Then a third of its volume of glycerin is added.

The fibers are macerated by boiling in a 1 per cent solution of sodium hydroxide and then thoroughly washed. The fibers are heated in the reagent on a slide. The mounting medium is concentrated glycerin.

After staining the fibers, it is well to wash them carefully in a glycerin solution (1 volume each of glycerin, water, and alcohol).

The flax fibers remain colorless, while the hemp fibers assume a greenish-blue color because the middle lamella is slightly lignified.

(X) *Determining Wool*: Various procedures have been suggested including:

Alternate (a):⁵² The following procedure may be used to distinguish wool fibers when they are dyed in various colors and mixed with cellulose fibers.

After placing the fibers on a slide glass, cover with a few drops of a 30 per cent solution of caustic soda and heat gently over a flame until boiling just takes place. Remove immediately, and examine under the microscope. Wool fibers appear very much

swollen and full of bubbles, whereas cellulose fibers remain unchanged.

Alternate (b):⁵³ Wool fibers may be determined quantitatively by treating the felt with 80 per cent sulfuric acid for 3 hours, whereupon the wool fibers remain as residue. A correction must be made if the felt contains mechanical wood fibers.

(XI) *Determining Rayon*: Various methods have been proposed, including:

Alternate (a):⁵⁴ A solution is prepared containing 1 per cent of picric acid and 0.2 per cent of "soluble blue 2B Extra" (Colour Index No. 702).

The sample is dipped for 3 min. in a cold or lukewarm solution. It is then washed well with cold water.

Acetate rayon fibers are stained strongly yellow. Cuprammonium rayon fibers are stained strongly blue. Viscose and nitrocellulose rayons remain colorless.

Alternate (b):⁵⁵ Precipitate a water suspension of methylene blue 2B (Colour Index No. 922) with about one-half the quantity of eosin yellow (Colour Index No. 768), stirring thoroughly to insure the completion of the reaction, separating the precipitate from the liquor by centrifugal force, followed by decantation, or by filtering (plain or with a Büchner funnel and suction flask).

Employing a white or light colored, *dry* sample of fiber for identification, cover it with a cold alcoholic solution of Wright's stain and bring in a few seconds just to a boil. Pour the reagent back into the bottle and wash the stained sample thoroughly with water.

Viscose rayon is stained blue; cuprammonium rayon is stained violet. Acetate rayon is also violet but partially disintegrated. Nitrocellulose rayon is stained deep blue.

Rayon samples when wet with water before staining all give a violet coloration. Hence, the dry condition when making the test.

(XII) *Identification of Fibers in Textiles*: Comprehensive methods of test have also been standardized for this purpose.⁵⁶

CHAPTER XXXV

EXAMINATION OF BITUMINOUS-SOLVENT COMPOSITIONS

Bituminous lacquers, cements, varnishes, enamels and japans are all characterized by the presence of a volatile solvent with a bituminous base, combined in the form of "vehicle." Depending upon whether or not the bituminous-solvent compositions contain a pigment or filler, they may be divided into two general classes, viz.:

- (1) Pigment or filler absent: including bituminous varnishes and japans, also certain bituminous lacquers and cements.
- (2) Pigment or filler present: including bituminous enamels, also certain bituminous lacquers and cements.

The first class consists of a vehicle made up of a solvent and base. The second consists of a pigment or filler combined with a vehicle, the latter similarly being made up of a solvent and base. The bituminous base may be composed of bituminous matter, with or without the presence of animal and vegetable oils or fats, resins or metallic dryers.

The following terms have been standardized for use in the paint, varnish and lacquer industry:¹

Accelerated Weathering Machine.—A machine used to determine the comparative durability of a finish in a shorter time than that required in actual service.

Air Dry.—Drying of a paint or varnish film dried at ordinary room temperatures (21–32° C.), unless a specific temperature is mentioned.

Alligatoring.—A defect in the paint or varnish film similar to checking, but having a much wider pattern. The top coats only are affected, and the cause is usually soft undercoats and hard-drying topcoats.

Baking.—The application of artificial heat to a paint or varnish film for stated periods of time and at stated temperatures.

Bending Test.—After applying a paint or varnish coating to a standard tin panel and allowing to dry, either air drying or baking,

the panel with the coated side up is bent quickly (within 2 sec.) over a mandrel of a specified diameter.

Bleeding.—When the color of a stain or other material works up into succeeding coats, imparting to them a certain amount of color, it is said to bleed.

Blistering.—The formation of bubbles on the surface of the film, either wet or dry. Usually due to moisture present behind the paint film. Sometimes the blisters disappear after the moisture has been removed.

Bloom.—A haze or clouded effect appearing on the surface of a dried enamel or varnish film, and affecting the gloss of the film.

Blushing.—This defect may occur during the drying period of lacquer films. Blushing is caused by the precipitation of a portion of the solid content of the material, which gives an opaque appearance. The two general types are "moisture" blushing and "gum" blushing. "Moisture" blushing is usually caused by high humidity. "Gum" blushing is usually caused by improper evaluation of the lacquer solvents, which results in a precipitation of some of the nonvolatile solids.

Bodied Linseed Oil.—Bodied oil is one that has been heat-treated so as to increase its "body" or consistency, and thus thicken it. Boiled oil on the other hand is only slightly thicker than raw oil.

Brittle.—A dried paint or varnish film that cracks readily in the bending test, is easily broken, and small pieces of which fly off under a knife blade.

Brushing Consistency.—A paint or varnish that is suitable, as received in the package, for easy application with a paint or varnish brush.

Chalking.—Chalking is a phenomenon of paint coatings manifest by the presence of a loose powder, coming from the film itself, at or just beneath the surface. This is detected on white paint by rubbing a piece of black velvet over it. Evaluated as slight, definite, and bad.

Checking.—A defect in the paint or varnish coating manifesting itself by slight breaks in the surface of the film. The break should be called a check if underlying paint or varnish coats are visible. "Visible checking" can be seen with the naked eye. "Microscopic checking" can be seen with a 10-power magnification. Evaluated as slight, definite, and bad.

Cloudiness.—When a nonpigmented material such as varnish, lacquer, or oil is not clear and transparent it is said to be "cloudy." This term is sometimes used in conjunction with blushing.

Color Floating.—The effect caused by one or more colors, other

than the original color, floating on the surface of the liquid paint in the package, or on the surface of the film during drying.

Color Retention.—The ability of a film of paint or varnish to show little change in original color after being exposed to the weather.

Cracking.—Cracking is a defect manifest in paint and varnish coatings by a break extending through to the surface painted. Where it is difficult to determine whether this is the case, the break should be called a crack if the underlying surface is visible. Evaluated as slight, definite, and bad.

Crawling.—A term used to describe the tendency of a wet finishing coat of paint or varnish to creep or crawl away from the dried undercoat. This results in a discontinuous film of the finishing coat.

Crow Footing or Crow's Footing.—A type of checking of a definite pattern, as indicated by the name, after a paint or varnish coating has been exposed to the weather. Also a type of crystallization on the surface of a varnish or paint film, after exposure to the gas test.

Disintegration.—The breaking down or failure of a paint or varnish film on exposure to weathering or some other agent.

Dry.—A term used to indicate when a film of paint or varnish has become completely solidified. The prepared panel is placed in a vertical position in a well-ventilated room but not in the direct rays of the sun. The atmosphere of this room must be free from products of combustion or laboratory fumes. The temperature of the room (unless otherwise specified) should be from 21 to 32° C. (70 to 90° F.). The film is tested at points not less than 2.5 cm. (1 in.) from the edges of the film. The paint or varnish is considered to have *dried hard* when the pressure that can be exerted between the thumb and finger does not move the film or leave a mark which remains noticeable after the spot is lightly polished. If rapid, light rubbing breaks the surface, the sample is considered not to have satisfactorily dried hard.

Eggshell Gloss, Eggshell Luster.—A term referring to the gloss of a dried paint film, the glossiness of which is between flat and semigloss, and resembles the glossiness of an eggshell.

Eggshell Flat.—The term refers to a flat oil paint that dries with little or no gloss except when viewed at a grazing angle. It refers to paints within the range "flat to eggshell."

False Body.—Refers to a paint having a thick consistency, but which brushes to a film of normal thickness. From the appearance in the package, the tendency is to add thinners to the paint. The result is that the thinned paint does not hide properly because the film is too thin.

Flaking.—The phenomenon of coatings manifest by the actual detachment of small pieces, generally irregular in shape, arbitrarily of an average diameter of less than $\frac{1}{4}$ in. (see "scaling").

Frosting.—A crystallizing effect on a paint or varnish film after being subjected to fumes, particularly oxides of nitrogen. As the name suggests, the film takes on a frosted appearance.

Gloss.—Gloss is the property of a surface by which it reflects light specularly.

Hiding Power.—The power of a paint as used to obscure a surface painted with it. This property is generally expressed in terms of square feet per gallon.

Leveling.—The flowing out of a paint, varnish, or enamel so that when the film is dry it shows no brush marks, orange peel effect, or ripples.

Lifting.—May result when one coat of finishing material is applied over another coat. This defect is manifest by wrinkling and is caused by the solvent action of a freshly applied coat on the binder of the undercoat. This defect is also known as "raising."

Livering.—The coagulation of a finishing material into a viscous, rubber-like mass.

Pulling under the Brush.—When insufficient material is used in the brush, or when the material is too heavy or too quick drying, there is a decided pull as the material is applied to the work.

Raising.—See Lifting.

Scaling.—An advanced form of flaking, and is arbitrarily chosen to be called scaling when the pieces of the coating that come off are of an average diameter greater than $\frac{1}{4}$ in. (see Flaking).

Self-lifting.—A special case of lifting in which both under coat and top coat are of the same material.

Self-raising.—See Self-lifting.

Semigloss.—This term refers to the glossiness of a finish and is between eggshell and high or full gloss.

Set to Touch.—An intermediate stage in the drying of a paint or varnish film which is reached when gentle pressure of the finger shows a tacky condition, but none of the coating adheres to the finger. The conditions for the test should be as stated under the term "Dry."

Skinning.—The tendency of paint, varnish, or enamel to form a surface layer of skin. This tendency is accelerated in an open or partially filled container. Some products show no skinning, others a slight skinning, and still others develop a thick, tough skin. Products should be stored in nearly filled closed containers. It is advisable to shake the container vigorously before putting it away.

Smooth.—A surface which does not feel rough to the touch.

Spreading Rate.—The amount of surface or area a given volume of paint can be spread over by brushing, spraying, etc. Usually expressed in square feet covered per gallon.

Sweating.—This term refers to a finishing material, but particularly to cabinet rubbing varnish, where the finish becomes more or less glossy after it is dull rubbed with pumice and water; usually due to the varnish not being hard dry before rubbing.

Whitening.—A change in the appearance of dried varnish, lacquer, or enamel films upon exposure to water, ranging from a bloom or blush-like effect to a chalky or milk-like appearance. This condition may or may not be permanent.

Yellowing.—The development of a yellow hue or the increase in saturation of a color of yellow hue. The term may also include darkening.

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT²

Test 84a. Specific Gravity. In the case of fluid materials, this shall be ascertained by means of a hydrometer (Test 7a) or a Westphal balance (Test 7b). Plastic cements shall be tested by means of a pycnometer (Test 7d). In either case, care should be taken to prevent evaporation of the solvent. The weight per gallon may be calculated from the specific gravity, by multiplying by 100 and dividing the product by 12.

The following method has been standardized for determining the weight per gallon:¹

In the case of liquid paints, weigh a clean, dry, 100-ml. graduated flask. Fill to the mark with the thoroughly mixed paint and weigh again. The increase in weight, expressed in grams, divided by 100, gives the specific gravity, which multiplied by 8.33 gives the weight in pounds per gallon. If one has a number of such determinations to make, it is convenient to have made a short cylinder of brass about 76 mm. high by 38 mm. in diameter with the inside of the bottom rounded, and having a capacity of 83.3 g. \pm 0.1 g. of water at 20° C. This cylinder is provided with a counterpoised weight. The paint is poured into the cylinder until it is completely full, the top leveled off with a spatula, and the full cylinder weighed to \pm 0.5 g. The weight in grams of the contents divided by 10 is the weight per gallon in pounds, and the weight per gallon in pounds multiplied by 0.12 is the specific gravity. In the case of pastes and

semipastes, use the brass cylinder method just described, with the added precaution of making sure that no air pockets are trapped in the material.

Test 84b. Viscosity. This is of value for purposes of factory control. If the material is sufficiently fluid, the Engler method (Test 8a) may be used, otherwise special viscosimeters may be used for the purpose, including the following:

Air-bubble Types: including Viscosity tubes, the Gardner-Holdt Bubble Viscosimeter, Collins Bubble Viscosimeter and the Steiner Bubble Viscosimeter.

Falling-weight Types: including the A.S.T.M. type,³ A. E. Robinson type, E. M. Symmes type and E. A. Lanz type.

Torsion Types: including the MacMichael Torsional Viscosimeter,⁴ Stormer Viscosimeter, Kämpf Viscosimeter, De Vilbiss Electro-Viscosimeter, Wolff-Hoepke Turbiviscosimeter, Thixotrometers of the New Jersey Zinc Co. type, Pryce-Jones type, Kewish & Wilcock type, Brookfield Synchro-Lectric Viscosimeter, etc.

Efflux Types: including Saybolt Universal Viscosimeter, Scott Viscosimeter, Westinghouse cup, Pratt & Lambert cup, Ford cup, Gottsch consistency cone, Parlin cup, Zahn cup, A.S.T.M. Consistency cup,⁵ etc.

Test 84c. Plasticity and Mobility. Various instruments have been described for testing these properties including the Bingham-Green Plastometer, the Gardner-Parks Mobilometer, the P.P.G. consistency tester, the Vacuum Plastometer (Gregory, Rassweiler and Lampert), the Hickson Penetration method, the Gardner Flowmeter, Hercules Capillary-tube Viscosimeter, Clarvov Consistometer, Kearsley-Roberts tester, Binney-Smith Flowmeter, Matthis Flowmeter, A.S.T.M. Cone penetrometer, etc.

Test 84d. Flash-point. This is of value in controlling the safety requirements. The Pensky-Martens Closed Tester (Test 17a) or the Tag Closed Tester (Test 17c) may be used for the purpose.

Test 84e. Brushability (Spreading Capacity and Workability). This test is of interest in determining the ease with which the material is applied and the surface area covered under normal working conditions. The material is spread on a clean surface of

a character on which it is intended to be used, by skilled workmen operating under normal conditions of temperature, light and humidity. The workability of the material is carefully noted and the area covered per gallon recorded. The following types of instruments have been proposed: Hart Brushability Tester,⁶ Baldwin-Gardner Tester,⁷ Floating-table Tester,⁸ etc.

Test 84f. Draining Test. This test has also been termed the "working viscosity test" and records the behavior of varnishes, japans and enamels when used for dipping purposes. It has been standardized as follows:⁹

A strip of sheet copper or brass $1\frac{1}{2}$ in. in width, 14 in. in length and 0.005 ± 0.0003 in. in thickness, shall be immersed in the varnish at a room temperature of approximately 25°C . (77°F .) up to a line previously drawn across the strip 1 in. from the top. The sheet shall be withdrawn at a slow and uniform rate (about 15 in. per minute), care being taken that the varnish is free from air bubbles. The specimen shall be permitted to drain thoroughly at room temperature while suspended in a vertical position. It shall then be dried or baked (according to the type of the varnish) until dry.

The thickness of the specimen in mils shall be measured at points 5.1 cm. (2 in.), 17.8 cm. (7 in.) and 30.5 cm. (12 in.), respectively, from the line to which the specimen was immersed. The thickness of each film in mils at the three points specified above shall be recorded. The difference between the thickness at the upper point (2 in.) and that at the lower point (12 in.) shall be taken as a measure of the variation in the film thickness caused by draining.

Test 84g. Drying Time. The times which elapse until the coating sets to the touch (i.e., ceases to be tacky), also when it sets to a firm, tough coating, furnish an indication of the speed in which the composition dries.

Method (I): For general use.—This test has been standardized as follows:⁴

Specimens for this test shall be films of varnish coated on thoroughly clean and smooth strips of copper $1\frac{1}{4}$ in. in width and 8 in. in length and 0.005 ± 0.0003 in. in thickness. The thickness of the cleaned copper strip shall be measured before the strip is dipped into the varnish.

The varnish shall stand in a covered tank for a sufficient length of time to be free of air bubbles, but not to exceed 1 hr., before the copper strips are dipped. The copper strips shall then be dipped once in the varnish at a temperature of approximately 25° C. (77° F.) and withdrawn slowly and uniformly (about 15 in. per min.). The consistency of the varnish shall be first so adjusted by trial that, when dry, the average thickness of the film of varnish on each side of the copper strip shall be between the measured limits of 0.0009 and 0.0010 in. The average thickness shall be calculated from the average of at least six thickness measurements taken in the middle half portion of the length of the strip. No thickness measurements shall be taken within $\frac{1}{8}$ in. of either edge of the strip.

NOTE.—It is recognized that the thickness cannot be measured with the precision stated but a close control of the thickness of the varnish film is desired. With the use of the micrometer, the actual average thickness may be expected to be the measured average thickness plus or minus 0.0002 in.

Specimens of air-drying varnish shall be dried in dust-free air at a room temperature of approximately 25° C. (77° F.). In the case of baking varnishes, six specimens shall be dipped and allowed to drain at a room temperature of approximately 25° C. (77° F.) until the varnish is set as indicated when the impression left on the surface by pressing lightly thereon with a finger at a point approximately 2 in. from the bottom will not become obliterated by further flow of the material. They are then to be dried in dust-free air in an oven at 105 to 110° C. (221 to 230° F.). At the end of the first 30 min., and again at the end of each 10-min. period thereafter, one specimen shall be taken from the oven and examined. In the case of slow-drying varnishes, this 10-minute period may be lengthened at the discretion of the operator.

NOTE.—The size of oven and the amount of ventilation have a considerable effect on the drying time of varnishes. For this reason the analytical type of electrically-heated oven should be used.

The varnish shall be considered dry when a piece of kraft paper 1½ in. in width and 6 in. in length and approximately 0.063 mm. (0.0025 in.) in thickness does not adhere to the varnish when it is pressed on the surface of the varnish for one minute by a cylindrical 1-lb. weight, 1 in. in diameter. The kraft paper shall be applied

in the vicinity of the center of the specimen and at right angles to it. The test shall be made at a temperature of approximately 25° C. (77° F.).

NOTE.—Certain types of varnishes dry with what is commonly known as a "tack"; but these types are rare. However, when testing them for drying time, it will be found that the kraft paper adheres to the varnish film and offers a certain amount of resistance to removal. On continued drying, the resistance to removal reaches constancy and is not changed by further drying. Therefore, the drying time is reported as the number of hours required to first reach constancy and the varnish should be reported as drying with a "tack."

The following alternate method of finding the drying time has also been standardized:¹⁰

Method (II): For varnishes, lacquers, and other nonpigmented coatings.—Flow the material on a 5 by 7-in. thoroughly cleaned, clear glass panel. Place the panel in a nearly vertical position in a well ventilated room, but not in the direct rays of the sun. Allow to dry under standard conditions. The film is tested at points not less than 2.5 cm. (1 in.) from the edges of the film.

Method (III): For pigmented paints, enamels, etc.—Brush 1 coat of the material on a standard glass panel and proceed as in method (II).

NOTE.—The material shall be considered to have set to touch when gentle pressure of the finger shows a tacky condition but none of the paint adheres to the finger. The material shall be considered to have dried hard when the maximum pressure that can be exerted between the thumb and finger does not move the film nor leave a mark which remains noticeable after the spot is lightly polished and the material shall be free from "after-tack."

The following additional procedures have been proposed for recording the drying time: Finger-touch method, Gardner Drying-time Meter,¹¹ Sanderson Drying-time Meter,¹² Paraffine Companies' Drying-time Machine,¹³ Parks Dry-O-Graph, Swinging-beam Tester,¹⁴ Wolff Spot-test,¹⁵ Blom Test,¹⁶ Federal Art Project Tester,¹⁷ etc.

Test 84h. Dry Film Thickness. The following method has been standardized for this test:¹⁸

A mechanical, dial type, thickness gauge suitably mounted on a rigid support shall be used. The painted specimen is suitably placed on the anvil with the foot of the dial resting gently on the paint film. The dial reading is noted. Raise the foot of the dial, carefully remove the paint by scraping with a knife blade, followed

by rubbing the spot with a wad of cotton (tied on the end of a rod) wet with acetone or extraction mixture, finally cleaning with fresh cotton, then lower the dial to the base and take a second reading. The difference gives the thickness of the film.

After the first dial reading is taken and during the time the paint is removed from the film support, *extreme caution* must be exercised in order that the painted panel is not perceptibly moved until after the second dial reading is taken.

Repeat the above procedure until a satisfactory number of readings have been taken from various areas of the surface.

Various instruments have been devised for the preparation of films of uniform thickness.¹⁹

Test 84i. Hiding Power. This is the property to obliterate any background upon which the composition may be spread under accurately regulated conditions. The methods usually employed include the use of the following instruments: Black-and-White Cryptometer,²⁰ Pfund Precision Cryptometer,²¹ Sward Rotary Cryptometer,²² Hallet Hidimeter,²³ Westinghouse Trans-O-Meter, British Research Station Transmeter,²⁴ Martens Photometer,²⁵ and others.²⁶

Test 84j. Color. Various instruments have been described for measuring the color of dried films, including the Visual Spectrophotometer, the G.E. Recording Color Analyzer, the Razek-Mulder Color Analyzer, the A.P.C. Spectrophotometer, the Appel-Hickson Spectrophotometer, the Munsell Universal Photometer, the Hess-Ives Tint Photometer, the Westinghouse Photo-Electric Color Matcher, the Watson Color Analyzer, the A.S.T.M. Spectrophotometer,²⁷ etc.

Test 84k. Gloss. Similarly, various methods have been proposed for measuring the gloss of dried films, including the Hunter Visual Glossmeter,²⁸ Hunter Portable Photo-electric Glossmeter,²⁹ Pfund Glossimeter,³⁰ Sheen-Meter,³¹ Ingersoll Glarimeter,³² Sward-Levy Gloss-comparator,³³ Detroit Club Meter,³⁴ Hunter Gloss-Comparator,³⁵ Gloss Inspection Lamp,³⁶ etc.

Test 84l. Light Reflection. The following instruments have been designed to measure the degree of light reflection of dried films: Hunter Visual Reflectometer,³⁷ Priest-Lange Reflectometer,³⁸ Hunter Multipurpose Reflectometer, Bausch & Lomb Opacimeter,

New Jersey Zinc Co. Photronic-cell Reflectometer, Ayres-Clewell Photometer,³⁹ the Nigrometer,⁴⁰ etc.

Test 84m. Hardness, Adhesion and Abrasion. Numerous devices have been proposed for ascertaining these properties, including the following:

Scratch Testers, such as the Lurie-Baily Hardness Tester,⁴¹ Graham-Linton Hardness Tester,⁴² Clemen Tester,⁴³ Wilkinson Pencil method,⁴⁴ Gardner Hardness Tester,⁴⁵ Du Pont Scratch Tester,⁴⁶ Schopper Hardness Tester,⁴⁷ Sheppard-Schmitt Scratch Dynamometer,⁴⁸ Michrocharacter Tester,⁴⁹ Parker-Siddle Scratch Tester,⁵⁰ Twisting-cork Tester,⁵¹ etc.

Pendulum Testers, such as the Walker-Steele Swinging-beam Tester,⁵² Sward Rocker Tester,⁵³ etc.

Intendation Testers, such as the Pfund Hardness Meter,⁵⁴ Imprint Resistance Tester, etc.

Distensibility Testers, such as the Gardner-Parks Distensibility Tester,⁵⁵ Rumpometer Tester,⁵⁶ Scott Tensile-strength Tester, Bell Laboratory Distensibility Tester,⁵⁷ Fuller Distensibility Test,⁵⁸ Mandrel tests,⁵⁹ Erichsen Film Tester,⁶⁰ etc.

Impact Testers, such as the Parlin-DuPont Impact Tester, Camp Impact Tester,⁶¹ Bell Laboratories Impact Tester,⁶² Hart Impact Tester,⁶³ the General Electric Method,⁶⁴ etc.

Adhesion Testers, such as the Cross-cut Adhesion Test,⁶⁵ Gardner Adhesion Test, Courtney-Wakefield Test,⁶⁶ Gelva Adhesion Test,⁶⁷ Schmidt Adhesion Test,⁶⁸ Chisel Adhesion Test,⁶⁹ Pebble Adhesion Test,⁷⁰ Liquid Wedge Test,⁷¹ Liquid Jet Test,⁷² etc.

Abrasion Testers, such as the Gardner Abrasion Test,⁷³ Bell Laboratories Abrasiometer,⁷⁴ Hercules Powder Co. Abrasiometer,⁷⁵ Bell Laboratories Rotating-disc Tester,⁷⁶ Parlin Abrasion Tester,⁷⁷ Camp Abrasion Tester,⁷⁸ Wolff Abrasion Tester,⁷⁹ Sward Abrasion Tester,⁸⁰ Wet Abrasion Test,⁸¹ A.S.T.M. Air-blast Tester,⁸² etc.

In addition to the foregoing, a road-service test has been proposed for traffic-paints.⁸³

Test 84n. Water Absorption. A specimen is spread on glass as in Test 84e and when dry, immersed in water at room temperature (approximately 70° F.) for 24 hrs. The film is then examined for adhesion, toughness, and in the case of insulating varnishes the dielectric strength is determined as in Test 84t.

Test 840. Resistance to Heat. This is of special value in the case of baking japans and insulating varnishes. This test has been standardized as follows:⁸⁴

Specimens shall be prepared by dipping into the varnish strips of thoroughly cleaned and smooth copper 8 in. in length, 1 in. in width, and 0.005 ± 0.0003 in. in thickness. The consistency of the varnish shall be adjusted previously by trial so that, when dry, the average thickness of the film of varnish on each side of the copper strip shall be between the measured limits of 0.0009 and 0.0010 in. The average thickness of each film of varnish shall be calculated from the average of at least six thickness measurements taken in the middle half portion of the length of the strip. No thickness measurement shall be taken within $\frac{1}{8}$ in. of either edge of the strip.

NOTE.—With the specified micrometer, the average thickness measured should be accurate to within plus or minus 0.0002 in. The close limits stated are intended for a close control of thickness which is necessary for this test.

Each specimen shall be dipped in the varnish at room temperature of approximately 25° C. (77° F.) and withdrawn slowly and uniformly (15 in. per minute). The varnish shall be allowed to dry in the air (if an air-drying varnish) or baked in an oven at 105 to 110° C. (221 to 230° F.) (if a baking varnish) until dry. As soon as the first coat of varnish is dry, and the specimen is at room temperature, it shall be dipped again in the varnish (readjusted to the original consistency) in the reverse direction so as to give a uniform thickness of coating. The second coat of varnish shall be allowed to dry in the same manner as the first coat. When the second coat of varnish is dry, the thickness of the specimen shall be determined. The measured average thickness of varnish on each side of the metal shall be between the limits of 0.0017 and 0.0021 in. The average thickness of each film of varnish shall be calculated from the average of at least six readings taken in the middle half portion of the specimen.

The specimens shall be placed in a uniformly heated oven, in which the temperature is maintained at 105 to 110° C. (221 to 230° F.). A specimen shall be removed at the end of the first 24 hrs. and every 24 hrs. thereafter.

NOTE.—Because of temperature variations between different levels in the oven, the specimen should be placed in the oven with the 8-in. dimension horizontal and the 1-in. dimension vertical. All specimens should be at the same level in the oven.

Each specimen, after heating, shall be tested at room temperature of approximately 25° C. (77° F.) by bending it through 180 deg. around a rod 0.32 cm. (0.125 in.) in diameter. The number of hours of baking of the specimen which first showed cracking of the varnish film shall be reported, together with the make and type number of the oven used.

NOTE 1.—The size of oven and the amount of ventilation have a considerable effect on the heat endurance of varnish films. For this reason the analytical type of electrically-heated oven should be used.

NOTE 2.—The temperature of the oven should be held as closely as possible to the mean (107.5° C., 225.5° F.). A difference of 5° C. (9° F.) in this temperature range, when continuously maintained, has a very considerable effect (approximately 25 per cent) on the life test of a varnish.

NOTE 3.—As the "grain" of the copper influences the result of the test, the heat endurance test specimens should be prepared by cutting the copper strips parallel with the direction of rolling.

Test 84p. Special Tests for Bituminous Enamels. See Tests 46b, 48a and 50.⁸⁵

Test 84q. Special Tests for Calking Compounds. The following methods of test have been standardized:⁸⁶

(1) *Rate of Hardening*: Two slabs of limestone 3½ in. square and ¾ in. thick are sealed on a metal spacer ¾ in. thick. One of the spaces is filled with the compound giving a joint ¾ in. wide, 1½ in. deep, and 3½ in. long. Penetration readings are made with a penetrometer similar to that used on bituminous materials, except the weight on the needle point is reduced to 12.5 g. The depth to which the needle penetrates into the compound in 5 sec. is recorded for three trials made along the centerline of the joint. After curing the joint in a horizontal position for 15 days at 70 ± 5° F. the skin is removed and three more penetration readings made. The hardening H is computed by the formula:

$$H = \frac{(P_1 - P_2)100}{P_1}$$

in which P_1 and P_2 are, respectively, the averages of the original and final penetrations.

NOTE.—In determining the original penetration (P_1) on very soft samples, the needle may sink to the bottom of the joint in less than 5 sec. In such cases it is

necessary to reduce the time of penetration to some feasible period and make the final penetration (P_2) for the same period.

Void spaces sometimes form within the compound and these cause erratic penetration readings. When the individual readings show large variations the number of readings should be increased accordingly.

(II) *Shrinkage*: Accessory materials required are: A brass ring (metal about $\frac{1}{32}$ in. thick) approximately $2\frac{5}{8}$ in. in diameter

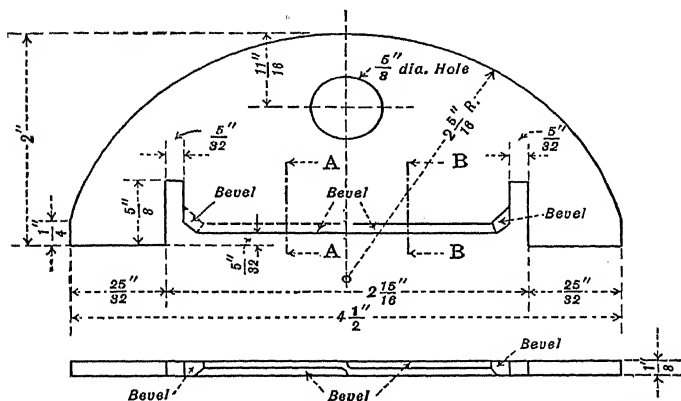


FIG. 377.—Tool for Leveling Calking Compound in the Shrinkage Test.

and $\frac{1}{2}$ in. wide, two ground glass cover plates 3 in. in diameter, a slab of limestone $3\frac{1}{2}$ in. square by $\frac{3}{4}$ in. thick, and a leveling tool for spreading the compound. (See Fig. 377.) One surface of each cover plate and both edges of the ring shall be ground with a fine abrasive on a flat metal plate until the ring will hold water when laid flat on the ground glass surface. The volume V of the ring is determined as follows: Weigh the ring and two ground glass plates to the nearest 0.01 g. The ring is then centered on one plate and poured full of water (temperature approximately 70° F.) and covered with the second glass plate, adding or removing water with a dropper until there are no air bubbles when the cover plate is centered. Overflow water or moisture on the outside ring or plates is removed and the weight of water required to fill the ring is obtained by weight differences. In this test it is sufficiently accurate to consider the volume of 1 g. of water as 1 ml., therefore

the volume of ring V is recorded as the difference between the second and first weights.

The shrinkage of calking compound is obtained as follows: Weigh the slab of limestone, the brass ring and a ground glass cover plate (all weights to the nearest 0.01 g.), and record the combined weight as W_1 . Then center the ring on the limestone slab and spread a $\frac{1}{8}$ -in. layer of the compound on the stone inside the ring, leveling with the tool to form good contact with ring and stone. The combined weight of the slab, ring, compound and cover plate (W_2) is then determined. The portion of the ring above the compound is then filled with distilled water at $70 \pm 5^\circ \text{F.}$ so there are no air bubbles when the cover plate is in place and the total weight (W_3) is determined. The volume of the compound is $V_c = V - (W_3 - W_2)$. The water is then poured off and the sample exposed to the air for 15 days, after which the weights W'_2 and W'_3 , corresponding to W_2 and W_3 , are determined. The shrinkage is computed by the formula:

$$S = \frac{(W'_3 - W'_2) - (W_3 - W_2)100}{V_c}$$

(III) *Slump*: A trough with rectangular cross section 1 in. deep, $\frac{3}{8}$ in. wide and 4 in. long is made of smooth sheet metal. This is filled with the compound and suspended in a vertical position for 24 hrs. at a temperature of $70 \pm 5^\circ \text{F.}$ and then 24 hrs. at a temperature of $122 \pm 5^\circ \text{F.}$ The amount of slump is measured from the lower end of the trough to the lowest point assumed by the compound.

(IV) *Bond*: Accessory materials required are a glass plate $3\frac{1}{2}$ in. square, a slab of limestone $3\frac{1}{2}$ in. square and $\frac{3}{4}$ in. thick and two brass rods 4 in. long and $\frac{5}{16}$ in. in diameter which have been planed off on two opposite sides to give a thickness of $\frac{1}{4}$ in. The rods are clamped or sealed to the stone slab with flattened sides in contact with the stone, each parallel to and about $\frac{1}{4}$ in. away from opposite edges of the slab. Thereupon a layer of the compound is spread over the surface of stone between the rods and built up to a thickness slightly greater than $\frac{1}{4}$ in. A thin layer of the compound is then applied to one surface of the glass plate in a strip about $2\frac{1}{2}$ in. wide so there are no air bubbles between the glass and

compound. This plate is then placed on top of the rods with the strip of compound in contact with the layer between the rods and forced down. The compound forced out of the joint shall be cleaned off and the specimen stored for 15 days at a temperature of $70 \pm 5^\circ \text{F}$. The test is then made by turning the rods 90° with a wrench so they will roll away from the compound, thus increasing the thickness of joint by 25 per cent. The area of separation from the glass may be estimated by superimposing, on the specimen, a glass or celluloid plate ruled into $\frac{1}{2}$ -in. squares.

(V) *Tenacity*: The test is made on the specimen used for determining shrinkage. After the shrinkage test is completed the brass ring is cut loose from the compound and removed. About half of the film of compound is loosened from the stone with a spatula and folded over on the remaining part, returned to the original position and the operation repeated until six folds have been made, so that for each fold the film is creased along the same line. Embrittlement or lack of tenacity is indicated by breaking or cracking of the film at the crease.

Test 84r. Resistance to Oil. This test has been standardized as follows: Specimens shall be prepared as described in Test 84o, "Resistance to Heat." The effect of oil on the varnish shall be determined by immersing the specimens in transformer oil at a temperature of 105 to 110°C . (221 to 230°F .) for 48 hrs. The specimen shall then be wiped with a piece of dry white cloth to determine whether or not the varnish has been affected.

NOTE.—Incipient disintegration of the surface of the varnish may sometimes be detected by examining the oil for turbidity. If a specimen of the used oil filtered through filter paper can be distinguished from an unfiltered sample of the used oil, when the two samples in identical containers are held in front of a diffused light, the oil is turbid.

Test 84s. Resistance to Acids and Alkalies. The dried film is subjected to the action of sulfuric acid sp. gr. 1.25 (about 33 per cent), nitric acid sp. gr. 1.12 (about 20 per cent), hydrochloric acid sp. gr. 1.09 (about 18 per cent), and sodium-hydroxide sp. gr. 1.15 for a period of 6 hrs., and the action noted.

Test 84t. Dielectric Strength. A method for testing insulating varnishes has been standardized, the details of which will be found in the original publication.⁸⁷ Other investigations have been

undertaken⁸⁸ to ascertain the relationship between the dielectric strength and the rust-inhibiting properties of bituminous-solvent compositions.

(B) ESTIMATION, RECOVERY AND EXAMINATION OF THE SOLVENT

Test 85. Estimation and Recovery of Solvent. Two methods have been adapted for this purpose, as follows:

(1) *Evaporation Method:* A method devised by A. L. Brown is rapid and gives accurate results, but does not recover the solvent for further examination.⁸⁹ Deliver 3-4 ml. of the well-mixed material (cements as well as paints of a heavy body should first be thinned to fluid consistency with a weighed quantity of pure benzol) from a 10-ml. pipette into a weighed glass flask of 50-ml. capacity, as rapidly as possible. Stopper the flask immediately, weigh, and dilute to the mark with pure benzol. Deliver exactly 10 ml. of the well-mixed material from the pipette upon a weighed ground-glass plate, 10 by 15 cm. and 1.5-3.0 mm. thick, supported in a level position. The diluted material should be flowed gradually on the plate, the object being to cover it entirely, without causing the solution to creep over the edges. It is recommended that 7 ml. be delivered first, and the remainder, a few drops at a time during the ensuing two minutes. The evaporation of the benzol will carry most of the solvent with it, and the film is so thin that the solvent will evaporate in one and one-half to two and one-half hours, the plate being weighed every half hour to follow the course of evaporation. Should the material contain a drying oil, the plate must be placed in an atmosphere of illuminating gas after the first half hour, replacing it after each weighing. The solvent has entirely evaporated when a constant weight is obtained. From this calculate the percentage of solvent by weight. An idea of the drying qualities of the film may be gained by placing the glass in a free circulation of air after the solvent is eliminated, and weighing it every hour as the film oxidizes, until it no longer *increases* in weight. If the coating has a tendency to dry unevenly, a weighed quantity of 50-mesh sea sand, previously dried and ignited, may be sifted over the paint in a very thin layer, but so the paint will be

visible between the grains of sand. This will insure a uniform evaporation of the solvent.

An alternate procedure has been standardized as follows:⁹⁰ The apparatus required consists of a chemical balance, a glass thermometer having a range of 0 to 150° or 200° C. and accurate within 1° C., a constant-temperature oven, a stoppered bottle and flat bottom metal drying dishes. Each metal drying dish shall have an inside diameter of approximately 2¾ in. (7 cm.) and a depth of approximately ⅝ in. (8 mm.).

NOTE.—A standard single-friction tin can cover having a diameter of 2⅜ in. is suitable for use as a drying dish.

The specimen shall consist of approximately 1.5 g. (1.35 g. to 1.65 g.) of the sample of varnish taken from the shipment. A portion of the sample of varnish shall be placed in a stoppered bottle or weighing pipette and weighed. Approximately 1.5 g. (1.35 g. to 1.65 g.) shall be transferred from the weighed stoppered bottle to a weighed drying dish which has previously been heated for 30 min. at 135° C. and cooled in a desiccator. The stoppered bottle with the remaining contents shall be weighed again. The exact weight of the specimen transferred to the drying dish shall be determined by difference. A total of three specimens shall be prepared from the contents of the stoppered bottle.

The specimens shall be placed in the constant temperature oven within 30 min. after preparation. The specimens shall be heated for a period of 3 hr. at the temperature designated, as follows:

Impregnating varnishes of the phenolic-resin type.....132 to 138° C.

Other types of insulating varnishes.....105 to 110° C.

NOTE.—In a gravity type of oven which depends upon the natural circulation of air for uniformity of temperature, only one shelf must be used for supporting the specimens, and the bulb of the thermometer for indicating the temperature shall be in close proximity to the specimens.

At the termination of the 3-hr. heating period, the specimens shall be removed to a desiccator for cooling to room temperature. Each specimen shall be weighed immediately upon removal from the desiccator. The percentage of nonvolatile matter by weight shall be calculated, as the ratio of the weight of the dried specimen to the weight of the specimen in the original state, expressed as a percentage.

The report shall include the following:

- (a) The type of varnish,
- (b) The oven temperature, and
- (c) The percentage of nonvolatile matter.

The following modified test is designed to determine the volatile matter by volume:

The purpose of this test is to determine the volume percentage of volatile matter (solvent) in a varnish, and to afford a means of arriving at the volume composition of the varnish with respect to volatile (solvent) and nonvolatile (base) constituents.

About 100 ml. of the varnish shall be distilled in accordance with method A.S.T.M. Designation: D 86. The distillation shall be "qualitative" instead of "quantitative"; that is, it is not necessary to record the volume of distillate recovered or the temperature of the initial boiling point or end point.

The distillation shall be continued until a suitable end point indicates that all the volatile matter (solvent) has been distilled off.

NOTE.—In the distillation of varnishes the end-point differs with the various types and compositions. In all cases, however, the rate of distillation slows down as the end-point is approached and, when the end-point is reached, acrolein (formed in the decomposition of the varnish base) is evolved and may be detected by its characteristic odor.

In addition to the evolution of acrolein, the following phenomena are suitable indicators of end-point:

(a) When the end-point of the solvent is considerably lower than the initial boiling point of the base, the end point of the distillation is indicated by the temperature reaching a maximum and then starting to fall consistently.

(b) When the end-point of the solvent and the initial boiling point of the base are too close, or overlap, the above temperature drop does not occur. The end-point of the distillation is then indicated by a sudden foaming up of the residual base in the distillation flask; or, if the distillation has been accompanied by foaming, the end-point is indicated by a breaking of surface tension with a consequent cessation of foaming.

Density: The density of the volatile matter (solvent) recovered by distillation shall be determined in accordance with the procedure described.

Volatile Matter by Weight: The difference between 100 per cent and the percentage of nonvolatile matter shall be calculated and recorded as the percentage of volatile matter by weight.

Volatile Matter by Volume: The percentage of volatile matter by volume shall be calculated as follows:

$$x = \frac{AC}{B}$$

where x = percentage of volatile matter by volume,

A = density of the original varnish as determined by test 84a

B = density of the volatile matter (solvent) recovered by distillation, and

C = percentage of volatile matter by weight.

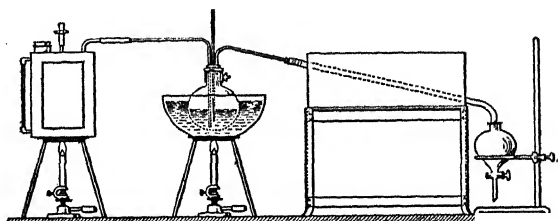
With proper care and attention to detail in making this test, differences occurring between different laboratories should not exceed 2 per cent for varnishes containing solvents which have end points not over 235° C. (455° F.), and should not exceed 4 per cent when the end point of the solvent is as high as 307.2° C. (585° F.).

(II) *Steam Distillation Method:* This has been standardized as follows:⁹¹ The bituminous mixture is distilled in a current of steam, the solvent is condensed and separated from the water. The steam-generator shall be made of either metal or glass, with a capacity of from 2 to 4 liters, suitable for continued use in the production of steam. If of glass, it shall be fitted with two outlets with suitable connections for rubber tubing. In the case of a metal generator, a large opening for filling and a water gage shall be additional parts of the apparatus. The generator shall be supplied with suitable pinch cocks or valves so that steam may be blown off to the atmosphere until the test is ready. The bath shall be of metal of sufficient capacity to permit immersion of the distilling flask to a depth of not less than 10 cm. Heat for the steam generator shall be supplied by a suitable gas generator or electric hot plate. The bath may be heated by any convenient means.

The distilling flask shall be a short ring-neck, round-bottom flask of 100-ml. capacity. It shall be fitted with a three-hole rubber stopper; with a steam distilling tube which will reach to within ½ in. (12.7 mm.) of the bottom of the flask and project from the top at a convenient distance for connection to the generator; a vapor outlet tube which extends from beneath the rubber stopper to a point sufficiently above the distilling flask that will permit convenient connection to the condenser; and with a thermometer. The steam tubing should be not less than 2 nor more than 4 mm. in in-

ternal diameter and the vapor outlet tube should be not less than 5 mm. in internal diameter.

The condenser shall consist of a $\frac{9}{16}$ -in. (14.29-mm.) outside diameter No. 20 Stubbs' Gage seamless brass tube, 22 in. (55.88 cm.) long. It shall be set at an angle of 75 deg. from the perpendicular and be surrounded with a cooling bath 15 in. (38.1 cm.) long, approximately 4 in. (10.16 cm.) wide by 6 in. (15.24 cm.) high. The lower end of the condenser tube shall be cut off at an



Courtesy A.S.T.M.

FIG. 378.—Assembly of Steam Distillation Apparatus.

acute angle, and curved downward for a length of 3 in. (7.62 cm.) and slightly backward so as to insure contact with the wall of the graduate at a point 1 to $1\frac{1}{4}$ in. (2.54 to 3.18 cm.) below the top of the graduate when it is in position to receive the distillate.

A separatory funnel having a capacity of not less than 500 ml. shall be provided. Accessories consist of suitable ring stands for supporting the steam generator, distilling flask, bath for distilling flask, separatory funnel, and a thermometer.

The apparatus shall be assembled as shown in Fig. 378. The steam-generator shall be filled with water and heat applied. The bath shall be filled with a high-flash-point oil and raised to approximately 140° C. (284° F.). Five hundred milliliters of the sample shall be weighed into the round-bottom flask. The steam-generator shall be connected to the steam delivery tube, the end of which shall be within $\frac{1}{2}$ in. of the bottom of the distilling flask. The outlet from the distilling flask shall be connected to the condenser and the separatory funnel placed in position at the outlet of the

condenser to receive the distillate. The end of the bub of the thermometer in the steam-distilling flask shall be placed within $\frac{1}{2}$ in. (12.7 mm.) of the bottom of the distilling flask.

When the temperature of the sample in the distilling flask reaches 130° C. the outlet of the steam generator shall be closed, thus forcing the steam to pass through the sample. The flow of steam shall be adjusted so that the distillate is collected at the rate of approximately 6 to 10 ml. per minute. The distillation shall be stopped when 100 ml. of the distillate contains not more than 0.5 ml. of solvent, as determined by measuring the amount of oil in 100 ml. of distillate. When the distillation is finished, the water shall be separated from the distillate and the distillate measured and retained for further tests, if required by the specifications.

In some cases, the distillate does not separate readily from the water, and this separation can be facilitated by the addition of sodium chloride, which will result in a sufficient difference in gravity to produce a clear separation of the two layers.

The results shall be reported in per cent by weight or volume as required by the specifications, based on the weight of the sample taken.

A rapid method has been described for determining the specific gravity of small volumes of solvent by a so-called "falling-drop method."⁹²

To correct the loss of distillate for water-soluble constituents, carefully weigh 100 g. into a 250-ml. flask and distil without steam over an electric stove. Continue the distillation until the residue in the flask reaches a temperature of 200° C. This gives somewhat lower results than the first method, but the distillate should be tested for water-soluble substances to correct the results obtained by the previous method. Turpentine dissolves to the extent of 0.3 g. for each 100 ml. of water condensed.⁹³

If the residue is to be used for further examination, any water introduced into it by the steam distillation may be removed by distilling it twice with ten times weight of a mixture of 3 parts benzol and 1 part alcohol.⁹⁴

Test 86. Examination of the Solvent. The solvent recovered from Test 85 should be tested for specific gravity, refractive index,

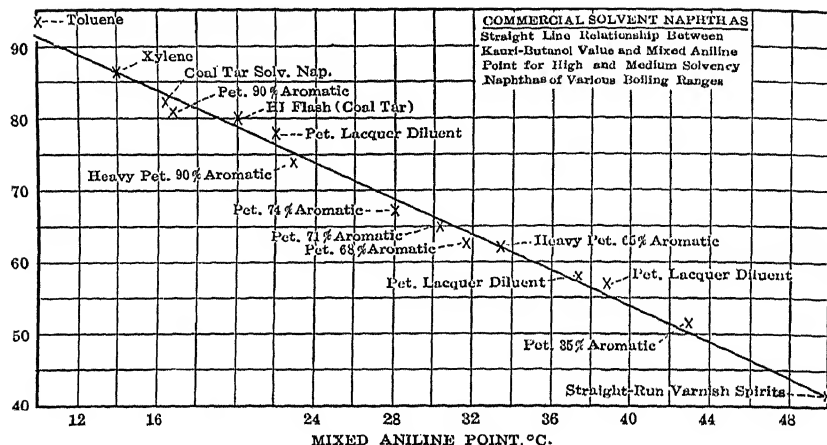


FIG. 379.—Mixed Aniline-point of Solvents.

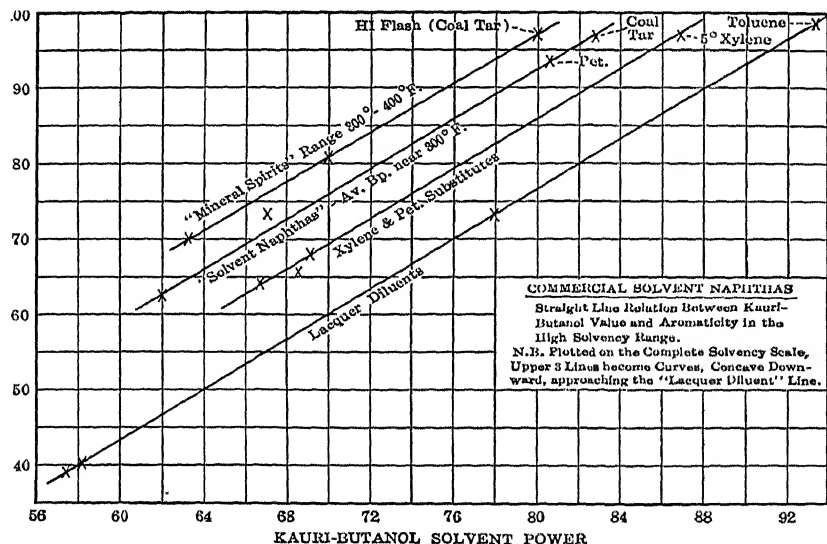


FIG. 380.—Kauri-Butanol Value of Solvents.

flash-point,⁹⁵ distillation range,⁹⁶ vapor pressure,⁹⁷ aniline point,⁹⁸ percentage unacted upon by concentrated sulfuric acid, etc., to assist in its identification.⁹⁹

Several tests have been proposed for measuring the "solveny" of petroleum naphthas,¹⁰⁰ including:

(a) Measurement of the aniline-point,¹⁰¹ which has been modified by the "mixed aniline-point" procedure,¹⁰² wherein the aromatic naphtha is diluted with an equal volume of a paraffinic naphtha whose aniline-point is 60° C.

(b) The kauri-butanol value, which covers the entire commercial naphtha solveny range, and is applicable to products of any degree of volatility.¹⁰³

A relationship between these two tests has been carefully worked out,¹⁰⁴ as illustrated in Figs. 379 and 380.

(C) ESTIMATION, RECOVERY AND EXAMINATION OF PIGMENT AND FILLER

Test 87. Estimation and Recovery of Pigment and Filler. Dilute 100 g. of the well-mixed material with 500 ml. of benzol in an 800-ml. stoppered flask. Either centrifuge or let stand in a warm place until the pigment or filler has settled, then carefully decant the supernatant liquid into a clean flask of large capacity. The pigment or filler is shaken up with 250 ml. more benzol, allowed to stand in a warm place until it settles, and the supernatant liquid decanted into the second flask. Repeat the treatment with benzol until the vehicle has been completely extracted from the pigment. The prevention of skin formation during this process may be attained by the addition of anti-oxidants such as phenol or hydroquinone (dissolved in ether),¹⁰⁵ which are subsequently expelled on heating the pigment and base. The combined extracts are allowed to stand quietly to recover any pigment that may have been carried over with the benzol, and then carefully decanted through a weighed Gooch crucible provided with an asbestos mat. The residues in the flask and on the Gooch crucible are washed with benzol as before, and combined with the balance of pigment or filler, which is then dried at 110° C. and weighed. The combined extracts are retained for further examination.

Test 88. Examination of the Pigment or Filler. The pigments or filler recovered from Test 87 are subjected to a qualitative or quantitative analysis for purposes of identification,¹⁰⁶ likewise to granulometric tests,¹⁰⁷ moisture and volatile constituents,¹⁰⁸ oil-absorption capacity,¹⁰⁹ etc.

(D) ESTIMATION, RECOVERY AND EXAMINATION OF THE BASE

Test 89. Estimation and Recovery of the Base. If no pigments or fillers are present, the base is recovered as described in Test 85, and its percentage by weight ascertained directly. If pigments or fillers are present, the combined extracts from Test 87 are distilled to a small bulk, transferred to a tared dish, and evaporated in an oven at 110° C. *exactly* to the calculated weight of the base, by subtracting the weights of solvent and pigment or filler from the original weight of material taken for examination. When oxidizable substances are present, the final evaporation should take place in an atmosphere of illuminating gas.

Test 90. Examination of the Base. The base will contain the bituminous constituents (with the exception of any "free carbon" associated with coal-tar pitch, or the like, which will be separated with the pigments and fillers), likewise animal and vegetable oils or fats,¹¹⁰ resins, and metallic bases and driers. It may be separated into its component parts in accordance with the procedure on page 1439, entitled: "Method of Analyzing the Separated Base."

The following is an outline of a method devised by the author for examining the dry films of lacquers, cements, varnishes, enamels, or japans which have been applied to surfaces of metal, wood, masonry, or prepared roofing. It often happens that none of the original material is available, and it becomes necessary to examine the paint after it has been applied to the object intended, and allowed to harden or oxidize in the air, either at normal or elevated temperatures. The method has been found to yield fairly accurate results.

Carefully scrape 50.00 g. of the lacquer or varnish film from the surface to which it has been applied, and avoid including any of

METHOD OF ANALYZING THE SEPARATED BASE

Dissolve 50 g. in 150 ml. benzol. Add 10 ml. dil. nitric acid (1 : 1) and boil under a reflux condenser for one-half hour to decompose any metallic soaps (i.e., driers, etc.). Add 150 ml. water, boil under reflux condenser, transfer to a separatory funnel, draw off the aqueous layer, boil with another 100 ml. water, and repeat if necessary until all the metals are removed.

Benzol Solution:

Distil to 100 ml., add 300 ml. of the saponifying liquid (Test 37c), boil under reflux condenser for one hour, and separate the unsaponifiable and saponifiable constituents as described in Test 37e.

Unsaponifiable Matter:

Examine a small portion by Test 41. If higher alcohols are present, separate the balance by Test 37f into:

Hydrocarbons:

Contain the bituminous substances (i.e., asphalt, coal-tar pitch, unsap. matter derived from fatty-acid pitch, etc.)

Examine by the methods included in Chap. XXXII.

Higher Alcohols, Etc.:

Contain cholesterol, etc., derived from wool grease, also the unsaponifiable constituents originally present in resins (4 to 8%).

Saponifiable Matter:

Separate the fatty and resin acids as described in Test 37g.

Fatty Acids:

Include acids derived from vegetable and animal oils or fats, also from fatty-acid pitch. (Note "A")

Resin Acids:

Include acids derived from rosin and the fossil resins. (Note "B")

Aqueous Layer:

Determine percentage glycerol by Test 37h. Multiply this by 10 to estimate per cent of vegetable or animal oils or fats (triglycerides) present in the original substance. (Note "C")

Aqueous Extract:

Contains the metallic bases as nitrates. Examine qualitatively and then quantitatively for lead, manganese, cobalt,¹¹¹ zinc, calcium, and magnesium.

(N. B.—The last three used for hardening rosin. The metallic driers should not be found by ignition, since the lead will be reduced to metal by the organic matter, and volatilize.)

Note "A"

The following means are used to distinguish between the fatty acids derived from oxidized vegetable or animal oils and fatty-acid pitch respectively:

	<i>Fatty Acids Derived From Vegetable or Animal Oils</i>	<i>Fatty Acids Derived From Fatty-acid Pitch</i>
Lactone Value (Test 37b).....	Less than 25.....	Greater than 25
K. and S. Fusing-point (Test 15a).....	Less than 80° F.....	Greater than 80° F.
Hardness at 77° F. (Test 9c).....	Less than 5.0.....	Greater than 5.0
Color in Mass (Test 1).....	Translucent yellow to brown.....	Opaque brown to black

Note "B"

Test qualitatively for rosin by the Liebermann-Storch reaction (Test 41). Fossil resins may be distinguished from rosin by determining the saponification, acid and ester values of the mixed resin acids. The following figures have been reported on the resin acids separated as described:¹¹²

	<i>Saponification Value</i>	<i>Acid Value</i>	<i>Ester Value</i>
Straight rosin varnish...	182-185	160-162	22-24
Rosin $\frac{1}{2}$; Kauri $\frac{1}{2}$ varnish	122-135	44-62	72-78
Rosin $\frac{1}{3}$; Kauri $\frac{2}{3}$ varnish	143.5	88	55.5
Straight Kauri varnish...	130	45	85
Untreated rosin.....	165-180	155-170	0-13
Untreated Kauri gum...	124	41	83

Other resins may be examined in a like manner, but unfortunately, figures are not at present available.

Note "C"

If this corresponds with the total saponifiable matter present, then fatty-acid pitch and resins are absent.¹¹³

the underlying surface.* From this point on the method is outlined in the following table:

METHOD OF ANALYZING DRIED FILMS

Boil 50 g. scrapings with 350 ml. of the saponifying liquid (Test 37e) under a reflux condenser for one hour. Add 300 ml. benzol-alcohol (1 : 1), boil, let settle and decant the supernatant liquid into a large flask. Repeat the treatment with benzol-alcohol (1 : 1) until most of the soluble constituents have been extracted, then combine the extracts and let stand quietly to recover any further settlings, which after decantation and washing are added to the main portion of the residue.

Benzol-Alcohol (1 : 1) Extracts:	Residue: Dry in an oven at 100° C., pulverize finely, transfer to a paper thimble and extract in a Soxhlet with benzol-alcohol (2 : 1) for 12 hours.		
Combine the benzol-alcohol (2 : 1) and (1 : 1) extracts, evaporate to a small bulk, and separate the unsaponifiable and saponifiable constituents as described in Test 37e.		Benzol-Alcohol (2 : 1) Extract:	Residue:
			Dry, ignite and weigh. This includes pigments and fillers, free carbon from tars or pitches, also any metallic dryers present (Note "A").
Unsaponifiable Matter: Examine as described.	Saponifiable Matter: Examine as described.	Aqueous Layer: Examine as described.	

Note "A"

This should be examined microscopically for fillers, and subjected to a qualitative or quantitative chemical analysis. Note that any chrome green, chrome yellow, Prussian blue, etc., are transposed by the alkali, and these, also lampblack or carbon blacks are decomposed on ignition, for which due allowance must be made.

Colorimetric tests have been devised for detecting various resins and synthetic resins as outlined in Table CLVI.¹¹⁴

* The blade of a safety razor held at right-angles to the surface scraped, and drawn across it slowly but firmly, has been found convenient for this purpose.

TABLE CLVI

CLASSIFICATION COLOR REACTIONS

Test	Procedure	Coloration	Observations
Liebermann-Storch (Storch-Morawski in Europe) for resin	Dissolve a small fragment of resin in hot acetic anhydride; cool. Add 1 drop of H_2SO_4 (sp. gr. 1.53) to soln. in spot plate or small porcelain crucible	Transient blue-violet Red Rose red	Indicated Resin Rosin, ester gum, etc. Coumarone, rosin adducts Cyclohexanone and cyclohexanone- CH_2O Vinyl resins
Halphen-Hicks for rosin (confirmatory)	Two reagents: A. 1 volume of phenol in 2 volumes of CCl_4 ; B. 1 volume of bromine in 4 volumes of CCl_4 . Add 1 to 2 cc. of soln. A to particle of resin in depression in porcelain spot plate. Stir. Fill adjacent cavity with soln. B. Cover with inverted watch glass and note color development in A	Bluish green Deep purple or deep indigo blue	Rosin
Resorcinol test for phthalates. Bradley modification of Hilde, Bleyberg, and Azziz test	Heat 0.25 to 1 g. of resin with 2 to 3 times this amount of pure resorcinol to boiling point of latter. Extract with boiling water, dilute, and render alkaline	Greenish-yellow fluorescence of fluorescein	Phthalates. Also applicable to phthalate plasticizers. Other dibasic acids may interfere
Phenol test for phthalates (phthalate confirmatory test)	Heat 1 g. of resin with 2 to 3 g. of pure phenol and 10 drops of concd. H_2SO_4 until formation of orange or brownish-orange melt. Cool, extract with boiling water, dilute, render alkaline	Red coloration of phenolphthalein in alkaline solutions	Phthalates
Gibb's indophenol for phenols (applicable to NaOH fusion residue or to destructive distillation products in aqueous solution)	Add 2 to 3 drops of 1% aqueous suspension of dibromquinone chloramide to 10 cc. of aqueous extracts of resin. Carefully neutralize by adding 0.1 N NaOH dropwise (to pH ca. 9.4)	Blue to wine Purple blue Faded purple	Phenol, cresols, and xylenols <i>p</i> - <i>tert</i> -Butylphenol and <i>p</i> - <i>tert</i> -amylphenol Reagent <i>p</i> -phenylphenol gives no color
Millon's reagent for phenols (metallic mercury dissolved in concd. HNO_3 , diluted with equal volume of water)	Add 4 drops of reagent to 5 to 10 cc. of aqueous soln. of products of destructive distillation. Shake, heat just to boiling	Deep red to brown Distinct lavender to purple; sediment in tube is edged with blue or purple on standing Rose	Phenol, cresols, and xylenols <i>p</i> -Phenylphenol <i>p</i> - <i>tert</i> -Butylphenol and <i>p</i> - <i>tert</i> -amylphenol Coumarone resin
Coumarone test for coumarone-indene resins	Dilute 1 cc. of 10% soln. of resin in $CHCl_3$ to 6 cc. with $CHCl_3$ and add 1 cc. of glacial acetic acid. Shake. Add 1 cc. of 10% soln. of bromine in $CHCl_3$, shake, allow to stand	Permanent red color	

CHAPTER XXXVI

EXAMINATION OF BITUMINOUS DISPERSIONS

(A) PHYSICAL TESTS OF THE FINISHED PRODUCT

Test 91. Method of Identification. The types of bituminous dispersions include the following: ¹

I. Quick-setting dispersions (i.e., those which set rapidly upon being mixed with mineral aggregate).

II. Slow-setting dispersions (i.e., those which set slowly upon being mixed with mineral aggregate). These in turn comprise:

(a) Semi-stable dispersions (i.e., those in which the breaking is retarded through the action of added "stabilizers").

(b) Stable dispersions, in which the setting results:

1. Mainly through evaporation of the water and to a slight extent by breaking.
2. Entirely through evaporation of the water content.

The foregoing types may be identified as follows: ² If necessary, dilute with distilled water until the content of bituminous matter is within 50 to 52 per cent by weight. Add two times the volume of 95 per cent ethyl alcohol.

If the bituminous matter remains in suspension = Type II-*b*-2.

If the bituminous matter separates as a gummy mass = Types I, or II-*a*, or II-*b*-1.

Dilute 10 ml. of the dispersion with 25 ml. distilled water and gradually add 20 ml. N/50 CaHCO_3 solution. Shake vigorously for 1 minute and heat to incipient boiling, thereby causing the bituminous constituents to settle out and adhere to the sides of the container. Cool and pour the liquid portion into a clean container, washing out the first container with two 5 ml. portions of distilled water. To the combined extracts, add slowly from a burette 0.1 per cent aqueous solution of Neptune Blue B. G. (manufactured

by I. G. Farbenindustrie A.-G.) until the brown color of the liquid turns to an olive green or greenish tone.

If less than 10 ml. of the dye solution is required = Type I.

If from 10 to 30 ml. of the dye solution is required = Type II-*a*.

If more than 30 ml. of the dye solution is required = Type II-*b-1*.

HOMOGENEITY

The following tests have been proposed:

Test 92*a*. Appearance Under Microscope. After diluting with a weak solution of NaOH (to prevent coagulation), transfer to a glass slide under a cover-glass, and measure the size of the particles under a magnification of 500 to 600 diameters. The particles should not exceed $5\ \mu$ in diameter. Typical fields of a well-prepared dispersion are illustrated in Figs. 381(*a*) and 381(*b*), the first under a magnification of 600 diameters, and the second a view of the same dispersion under a magnification of 1200 diameters.

Test 92*b*. Sieve Test. Coarse particles and lumps may be ascertained by the following standard method:³

The following apparatus and reagents will be required:

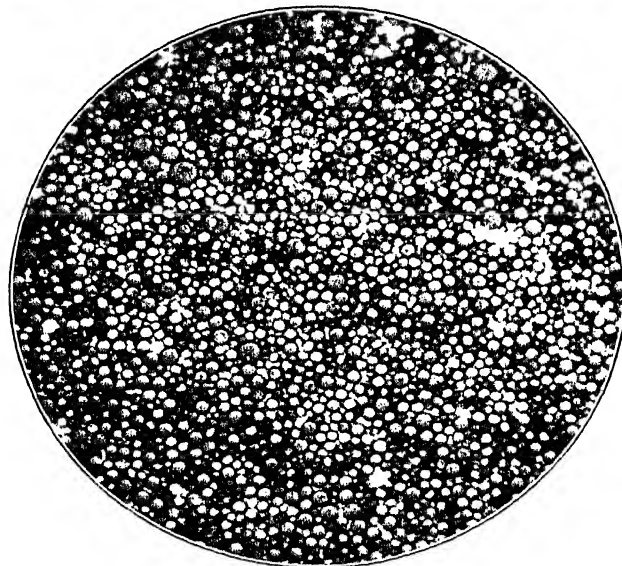
(*a*) Sieve: A No. 20 sieve, having a 3-in. frame, of the U. S. Standard Sieve Series.

(*b*) Pan: A tin box cover or shallow metal pan of appropriate size to fit over the bottom of the standard sieve.

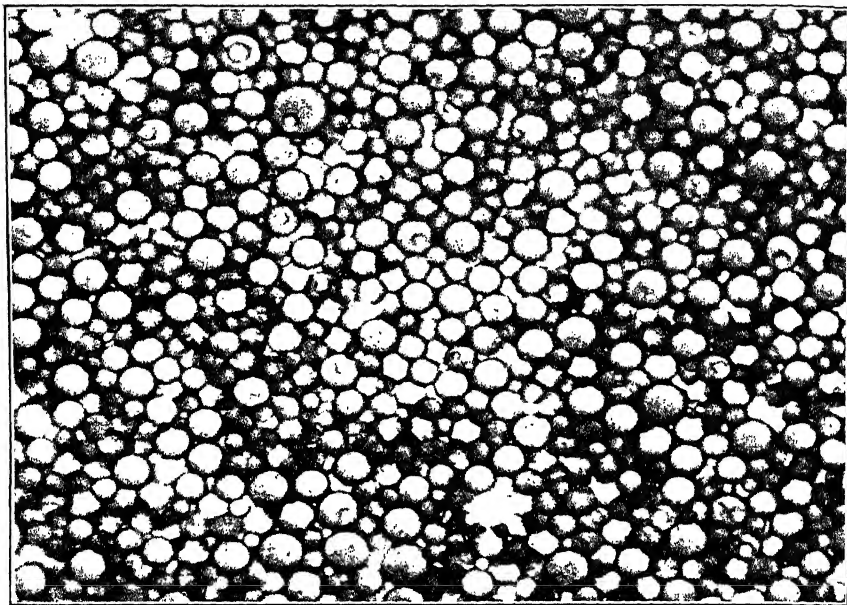
(*c*) Sodium Oleate Solution (2 per cent): Prepare a 2 per cent solution of pure sodium oleate in distilled water.

The weight of the sieve and pan shall be recorded and the wire cloth of the No. 20 sieve shall then be wet with the sodium oleate solution (2 per cent). Exactly 1000 g. of the emulsified asphalt shall then be weighed and poured through the wire sieve, the container and the residue on the sieve being washed thoroughly with the sodium oleate solution until the washings run clear. The pan shall then be placed under the sieve and heated for 2 hr. in a drying oven whose interior temperature is 220° F. (105° C.), then cooled in a desiccator and weighed.

The total weight of the sieve, pan, and residue in grams, less the combined tare weight of the sieve and pan, is the weight of the



(a)



(b)

FIGS. 381 (a) and 381 (b).—Typical Bituminous Dispersions in Water.

residue by the sieve test. The percentage of residue in the emulsion shall be calculated on the basis of this weight.

Test 92c. Settlement Test. This has been standardized as follows:³

The following apparatus will be required:

(a) **Cylinders:** Two glass cylinders of 500-ml. capacity with pressed or molded glass base and cork or glass stoppers. The outside diameter shall be 5.0 cm. \pm 0.5 cm. and the cylinders shall be graduated at each 5-ml. interval to the 500-ml. mark.

(b) **Glass Pipette:** A syphon, glass tube pipette, 60-ml. capacity, form optional.

A 500-ml. sample, representative of the emulsion, shall be placed in each of two glass cylinders. The cylinders shall be stoppered air-tight and stood aside unmolested, at laboratory air temperature for 5 days. After standing for this 5-day period, approximately the first 55 ml. of emulsion shall be removed by means of the pipette or syphon from the top of each cylinder without disturbing the balance of their contents. Exactly 50 g. of each of the two samples, after each has been thoroughly mixed separately, shall be weighed into separate 600-ml. low-form glass beakers and the asphaltic residue determined by evaporation at 325° F. (163° C.) for 3 hr. in the apparatus described in Test 16.

After removal of the first sample, approximately the next 390 ml. shall be syphoned off from each of the cylinders. The residue remaining in the cylinders shall be mixed thoroughly and exactly 50 g. shall be weighed out from each of them and the amount of asphaltic residue (all sediment, if any, included) shall be determined by evaporation as described above for the two top samples.

The numerical difference between the average percentage of asphaltic residue from the two top samples and the average percentage found in the two bottom samples shall be recorded.

Test 92d. Stability on Aging. The dispersion is allowed to stand in a tightly closed container and retested as in 92b and 92c at the end of 1, 3 and 6 months respectively, to ascertain whether it has a tendency to separate or "reverse" upon standing.

Test 92e. Determination of pH Value. Bituminous emulsions may be tested for their pH value as follows:^{3a} Place 2 drops of the emulsion on a white porcelain tile and cover with a slip of filter-

paper. When the upper surface of the paper is moistened by the emulsion, add a drop of indicator B.D.H.-4.11 close to the wet spot and note the color of the intersecting arcs of the indicator and the emulsion liquid. The pH value may be estimated from the observed color. Where greater accuracy is desired, repeat the test with different indicators which change color at the pH value determined by the first test.

Test 93. Viscosity. The viscosity determination shall be made at 77° F. (25° C.) and shall be expressed in seconds, Saybolt-Furol, being the time in seconds for the delivery of 60 ml. of emulsion. While the Saybolt-Furol viscosimeter is not used for petroleum products and lubricants when the time of flow is less than 25 sec., this instrument is satisfactory for testing emulsified asphalt when the time of flow is not less than 20 sec.

The sample shall be stirred thoroughly, without incorporating bubbles in it and then poured into a 4-oz. bottle. The bottle shall then be placed in the water bath at 77° F. (25° C.) for 30 min. and the sample then mixed in the bottle by inverting several times, slowly enough to prevent bubble formation. The sample shall then be poured into the viscosity tube through a 20-mesh strainer, allowing a small portion to flow through the outlet tube to waste. The cork shall then be placed in position, the tube filled and without again stirring the sample the viscosity shall be determined as described in Test 8b.

DEMULSIBILITY

This indicates the rapidity with which the dispersion "breaks" during use. It has been shown that there is no relationship between the results obtained by the demulsibility test and the "rate of break" of the emulsion when used with different character of aggregates.⁴ The demulsibility may be ascertained in accordance with the following procedures:

Test 94a. Calcium-chloride Test:⁵ This test has been standardized as follows:³

The following apparatus and reagents will be required:

- (a) Sieves: Three No. 14 sieves of the U. S. Standard Sieve Series, of iron wire cloth, unframed, approximately 5-in. square.
- (b) Beakers: Three glass beakers of 600-ml. capacity each.

- (c) Glass Rods: Three metal rods, rounded ends, approximately $\frac{5}{16}$ in. in diameter.
- (d) Burette: A 50-ml. glass burette graduated in 0.1 ml.
- (e) Calcium Chloride Solution (0.02 *N*).
- (f) Calcium Chloride Solution (0.10 *N*).

The percentage of residue shall be determined by distillation as described in Test 98.

The weight of each assembly of beaker, rod and sieve shall be recorded. Exactly 100 g. of the emulsified asphalt shall be weighed into each of three 600-ml. tared beakers. Over a period of approximately 2 min., 35 ml. of 0.02 *N* CaCl_2 solution (if quick-setting emulsion is being tested) or 50 ml. of 0.10 *N* CaCl_2 solution (if mixing type emulsion is being tested) shall be added to each beaker from a burette. While adding the solution of CaCl_2 , the contents of the beaker shall be stirred continuously and vigorously, kneading lumps against the sides of the beaker to insure thorough mixing of the reagent with the emulsion. This operation shall be performed after bringing the weighed sample of emulsion and the reagent to the standard temperature of $77 \pm 1.0^\circ \text{F}$. ($25 \pm 0.5^\circ \text{C}$.).

One of the wire sieves shall be fitted over a beaker or other suitable vessel and the mixture of emulsion and reagent poured through the sieve. The beaker, containing the sample and glass rod, shall be rinsed with distilled water. All lumps shall be kneaded and broken up, and the washing of the beaker, rod and sieve shall be continued until there is no longer any appreciable color imparted to the wash water. After washing as directed, the beaker, rod and sieve used in each individual test shall be placed in a drying oven and dried at 325°F . (163°C .) to constant weight.

The total weight thus obtained less the total tare weight of the beaker, rod and sieve shall be the weight of the residue by the demulsibility test. The ratio of the average weight in grams from three tests of each individual sample of emulsified asphalt, *A*, to the weight in grams of residue per 100 g. of emulsion, *B*, obtained in the test for residue by distillation described in Test 98 multiplied by 100, shall be recorded as the percentage of demulsibility of the sample tested:

$$\text{Percentage Demulsibility} = \frac{A}{B} \times 100$$

Test 94b. Ferrous-sulfate Test. This test is claimed to be more severe than the Calcium-chloride Test, and is determined as follows: 50 g. of the dispersion are weighed into a 250 ml. beaker, diluted with 25 ml. distilled water and 10 ml. of a freshly prepared 10 per cent $\text{FeSO}_4 \cdot 7\text{H}_2\text{O}$ solution added during a period of approximately 2 minutes, the contents of the beaker being continuously stirred with a glass rod. The solution is diluted to 100 ml. and permitted to stand for 24 hours, when an additional 100 ml. of distilled water are added. The contents of the beaker are then drained through a 40-mesh sieve and the unbroken emulsion in the beaker and on the rod is rinsed through the sieve with water until there is no appreciable discoloration of the rinsing water. The beaker, rod and sieve are permitted to dry at room temperature and weighed every 24 hours until constant weight is attained. The weight of residue deposited on the beaker, rod and sieve is recorded as an index of the demulsibility.

Test 94c. Behavior with Aggregate ("Coating Test"). This test has been standardized in the following manner: "

NOTE.—This test is applicable only for emulsions containing a base of asphalt of semi-solid consistency. It is not applicable to the so-called quick-setting type of emulsions.

The following apparatus and reagents will be required:

(a) Screens: A standard $\frac{3}{4}$ -in. screen and $\frac{1}{4}$ -in. screen conforming to the requirements of the Standard Specifications.

(b) Spatula: A steel spatula or its equivalent, blade approximately 8 in. in length.

(c) Dish: A round-bottom, iron dish or a kitchen saucepan, approximately 1-qt. capacity.

A supply of reference stone (hard limestone, trap rock, or other type) which has been washed with water and dried before using. The grading of this stone shall be such that it will all pass through a standard $\frac{3}{4}$ -in. screen and not more than 5 per cent will pass through a $\frac{1}{4}$ -in. screen.

NOTE.—Each laboratory shall select its own reference stone supply, the source of which is not apt to change. This is to obviate rapid changes in the character of reference stone used in any one laboratory.

Exactly 465 g. of the washed and dried graded stone shall be weighed and placed in the metal pan. A 35-g. sample of the emulsion shall then be added to the stone in the pan and mixed vigorously with the spatula for 3 min.

Record whether or not there is appreciable separation of the asphaltic base from the water of the emulsion and whether or not the stone is uniformly and thoroughly coated with the emulsion.

The foregoing demulsibility test will indicate whether the emulsions are of a slow-setting type or a quick-setting type.⁷ Slow-setting emulsions, otherwise termed "mixing emulsions," must be capable of dilution with water, and when used for coating aggregates by machine or hand-mixing methods must permit a uniform distribution and of handling and manipulation of the mixture incident to construction. Quick-setting emulsions, otherwise termed "penetration emulsions," must coalesce or demulsify as soon as they come in contact with the aggregate when they are spread over the pavement already laid in place. Slow-setting emulsions contain considerable quantities of dispersing agents, and set largely by evaporation of the water present. Quick-setting emulsions contain either very small quantities of the dispersing agent, or a small quantity of a previously formed quick-setting emulsion.⁸

Test 94d. Behavior with Portland Cement. The following procedure has been standardized:³

This method of test is intended for determining the amount of coagulation when portland cement is mixed with the slow-setting type of asphalt emulsions for fine aggregate mixes. This test is not applicable when 2 per cent or more coagulation is anticipated.

The apparatus shall consist of the following:

(a) Sieves: A 177-micron (No. 80) sieve and a 1410-micron (No. 14) sieve, made of iron wire cloth having wire diameters and openings conforming to A.S.T.M. Designation: E 11.

(b) Dish: A round-bottom iron dish or a kitchen saucepan of approximately 500-ml. capacity.

(c) Stirring Rod: A steel rod with rounded ends, approximately $\frac{1}{2}$ in. in diameter.

(d) Graduate: A 100-ml. graduated cylinder.

The high-early-strength portland cement used in the test shall conform to the requirements for type III of the Standard Specifica-

tions for Portland Cement (A.S.T.M. Designation: C 150) and shall have a minimum specific surface area of 1900 sq. cm. per gram.

Dilute the emulsion to be tested with distilled water to a residue of 55 per cent as determined by either distillation, or evaporation for 3 hrs. at 163° C.

Sieve a portion of the cement through the 177-micron (No. 80) sieve. Weigh 50 g. of the cement passing the 177-micron sieve into the iron dish.

Add 100 ml. of the diluted emulsion to the cement in the dish and stir the mixture at once with the steel rod, using a circular motion, making 60 complete revolutions during 1 min. Immediately at the end of the 1-min. mixing period, add 150 ml. of distilled water and continue the stirring for 3 min. The ingredients and apparatus shall be maintained at a temperature of approximately 25° C. during the mixing period.

Pour the mixture through the tared 1410-micron (No. 14) iron sieve, of approximately 3 in. diameter and rinse by pouring distilled water from a receptacle held at a height of approximately 6 in. Place the sieve in a tared shallow pan, heat at 163° C. in an oven until dry, and weigh.

Report the weight in grams of the material retained on the sieve and in the pan as the percentage of the emulsion broken.

BEHAVIOR WITH WATER

Test 95. Miscibility with Water. This test is performed as follows:^s

NOTE.—This test is not applicable to the so-called quick-setting type of emulsions.

To about 50 ml. of the emulsion, shall be gradually added about 150 ml. of distilled water, stirring the mixture while adding the water. The temperature is not important but should be between 21 and 25° C. (70 and 77° F.). The mixture shall be allowed to stand for 2 hr. and then examined for any appreciable coagulation of the asphalt content of the emulsion.

A modified miscibility test may be performed as follows:

This method of test covers the procedure for determining the miscibility with water of medium-setting and slow-setting types of

asphalt emulsions. This test is not applicable to the quick-setting type of asphalt emulsions.

The apparatus shall consist of the following:

- (a) Graduate: A 50-ml. graduated cylinder.
- (b) Beaker: A 400-ml. Griffin low-form glass beaker.
- (c) Glass Tubes: Three glass tubes, 7 mm. in outside diameter, 5 mm. in inside diameter, and 15 cm. in length, fitted with suitably bored No. 8 corks, adjusted as described.
- (d) Supporting Strip: A strip of metal or wood, approximately 15 cm. in length, 2.5 cm. in width, and 0.5 cm. in thickness, with a hole 10 mm. in diameter in the center.
- (e) Crucibles: Three 15 or 25-ml. porcelain crucibles, or three 30-ml. beakers of heat-resistant glass.*
- (f) Oven: A constant temperature oven as described in A.S.T.M. Designation: D 6.
- (g) Balance: An analytical balance accurate to 0.1 mg.

Adjust the position of the corks on the glass tubes by measuring 200 ml. of distilled water at 20 to 25° C. into the 400-ml. beaker, placing the supporting strip across the top of the beaker, inserting a tube through the hole, and adjusting the position of the cork so that when the tube is supported by the cork resting on the supporting strip, the lower end of the tube is immersed in the water to a depth of 1 cm. below the surface. In the same manner, adjust the second and third tubes so that the depth of immersion is 2.5 and 4.6 cm., respectively.

NOTE.—Due to slight differences in height and diameter of 400-ml. beakers as obtained commercially, it may be necessary to readjust the tubes when used in different beakers. In any event, the third or bottom tube shall project into the emulsion so that the tip is within 1 to 1.5 mm. of the bottom of the beaker.

Measure 50 ml. of the emulsion at a temperature of 20 to 25° C. into the graduated cylinder and transfer to the 400-ml. beaker. Wash the graduate with three 50-ml. portions of distilled water at 20 to 25° C. and add the washings to the beaker, bringing the final volume to 200 ml. Stir the emulsion and water with a glass rod until uniformly mixed, cover the beaker with a watch glass, and allow the mixture to stand undisturbed for 2 hrs.

Weigh the three crucibles or 30-ml. beakers, and a watch glass for each, to the nearest 0.1 mg. After the diluted emulsion has stood for 2 hrs., remove the watch glass and place the supporting strip across the top of the 400-ml. beaker. Take a sample of ap-

* Pyrex glass is very satisfactory for this purpose.

proximately 1 g. from the top layer and transfer to one of the crucibles or beakers, using the first or 1-cm. depth tube as a pipette. Close the top of the tube with the finger, insert the tube to the proper depth, remove the finger while the emulsion rises in the tube, and then replace the finger on top of the tube so that when the tube is removed its contents of emulsion will be pipetted from the beaker. After removal, wipe off the adhering liquid on the outside of the tube with filter paper before transferring the sample to the crucible. In like manner, take samples from the middle and bottom of the diluted emulsion, using the second and third tubes, respectively. Weigh the crucibles with their accompanying samples of emulsion, and determine the weight of each of the three samples by difference. Cover the crucibles with watch glasses to retard evaporation.

Remove the watch glasses from the crucibles and place the samples in the oven at 163° C. for 2 hrs., then remove, cool, and weigh.

Calculate the percentage of asphalt residue in the top, middle, and bottom levels. Report the maximum numerical difference in percentage of asphalt content between any two of the three levels.

Test 96. Effects of Freezing.⁹ This test has been standardized as follows:³

Approximately 400 g. of the emulsion shall be placed in a clean metal container, such as a 1-pt. press-top tin. The emulsion in the closed container shall be exposed to a temperature of 0° F. (-17.7° C.) for twelve consecutive hours. At the expiration of the freezing period, the emulsion shall be permitted to thaw by exposure of the container to the temperature of the laboratory.

After the first operation of freezing and thawing, the procedure shall be repeated twice, so that the emulsion will have been subjected to three cycles of freezing and thawing.

After the third cycle, the emulsion may be homogeneous, or may have separated into distinct layers which cannot be rendered homogeneous by stirring at laboratory temperature.

The result of this test shall be reported as either "Homogeneous" or "Broken."

Test 97. Resistance to Water after Setting.

(1) *Without Aggregate:* A slight excess of the emulsion (thinned with water to the consistency in which it is recommended

to be used) is poured into a clean porcelain dish which by suitable manipulation is caused to flow over the entire inner surface. The excess is then allowed to drain out. The dish is then stood upright at room temperature and observed from time to time until the film has dried, which may generally be ascertained when it turns from a brown to a black color. The time of drying is recorded, whereupon the dish is maintained at room temperature for twenty-four hours. At the termination of this period it is filled with distilled water and examined at the end of one, two, four and eight hours respectively, to determine whether or not the film is softened or dissolved by the water.

The following procedure has been standardized for testing the films deposited from bituminous emulsions:¹⁰

A. *Resistance to Flow Under Heat (Slide Test)*. The slide test is applicable to bituminous emulsions or reinforced emulsions intended to be applied at the rate of not less than 3 gal. per 100 sq. ft. and on substantially vertical surfaces.

The apparatus shall consist of the following:

(a) A brass mask approximately $\frac{7}{16}$ in. in thickness with a rectangular opening 4 by 4 in.

(b) Unglazed ceramic tiles white, nonvitreous, dust pressed body with an absorption range of 15 to 18 per cent (determined in accordance with the Standard Methods of Sampling and Testing Structural Clay Tile, A.S.T.M. Designation: C 112) approximately 6 by 6 in. by $\frac{3}{8}$ to $\frac{1}{2}$ in. in thickness, or of sufficient size to accommodate the opening of the mask.

The tile shall be immersed in distilled water for at least 10 min. and the excess water removed immediately before application of the emulsion.

The sample of emulsion shall be thoroughly stirred. The brass mask shall then be applied to the smooth face of the tile and the emulsion spread over the area of the tile within the mask opening. The excess emulsion shall be doctored off with a flat scraper so that the film after drying shall be not less than 0.04 in. (1 mm.) in thickness.

The coated tile shall then be dried to constant weight in an atmosphere of low relative humidity at room temperature and

weighed every 24 hrs. A film shall be considered dry when the loss in two successive 24-hr. weighings is not greater than 0.1 g.

NOTE.—Drying may be hastened by placing the specimen in a current of air from an electric fan.

After drying and removing the brass mask, the coated tile shall be suspended vertically in the center of an air oven maintained at $176 \pm 5^{\circ}$ F. ($80 \pm 3^{\circ}$ C.). The internal dimensions of the oven shall be not less than 12 by 12 by 12 in. An electrically heated oven is recommended. A thermometer shall be inserted in the oven to such depth that its bulb will be in line with the center of the specimen. The tile shall be maintained at the prescribed temperature for exactly 2 hrs. Any sliding shall be determined by a reference line placed originally on the tile, coincident with the lower edge of the dried film.

B. Resistance to Water Action. The purpose of this test is to determine the ability of the dried film to retain its adhesion and to resist re-emulsification after immersion in water.

The film to be tested shall be prepared as described.

After drying, a ring approximately 2 in. in diameter and 1 in. in height shall be pressed into the surface of the coated tile. The ring shall be filled with tap water and the test specimen maintained at room temperature between 70 to 90° F. (21 to 32° C.). After 24 hrs. the character of the bituminous film shall be noted by cutting and attempting to lift a portion of the submerged film with a knife or teasing needle. Adhesion or bond shall be determined by making intersecting cuts with the knife or needle and lifting the cut film at the point of intersection. Re-emulsification is indicated if the water becomes darkened by rubbing the submerged surface of the uncut film lightly with a rubber policeman.

(II) *When Mixed with Aggregate:* Clean, rounded river gravel of uniform quality from a source permitting of duplication for future work should be selected. Two 1000-g. samples passing a $\frac{1}{2}$ -in. and retained on a $\frac{1}{4}$ -in. screen are placed in wire baskets and dried to a constant weight in an oven at 125° F. The baskets are immersed for two minutes in the emulsion to be tested. The emulsion and aggregate are maintained at 77° F. during the test and the room at 77° F. at a relative humidity of about 50. After

removal from the emulsion, one basket is allowed to drain exactly 30 min. and is then washed with tap water for 15 min., by allowing a stream of water to flow through a $\frac{1}{4}$ -in. hose at the rate of 1 gal. per min. until the water comes through clear. The basket and contents are dried to a constant weight in an oven at 120° F. The increased weight due to the deposited asphalt is recorded. The other basket is dried to a constant weight in the same manner, without spraying with water. The asphalt remaining on the aggregate in the washed basket is calculated as a percentage of the asphalt deposited on the aggregate in the unwashed basket.

(III) *When Mixed with Soil:* A mixture of soil "stabilized" with asphalt emulsion is molded into cylinders 2 in. in diameter by 4 in. high, at a moisture content which will produce dense specimens. These are then dried at 140° F. and recompressed under a load of 3000-lb. per sq. in. They are then placed in shallow pans, the bottoms of which are covered with standard Ottawa sand, over which is laid a heavy cotton or flannel cloth. The lower half of the cylinders is immersed in water, and at the end of 7 days they are reweighed to ascertain the water-absorption, which is recorded in per cent.¹¹

(B) SEPARATION OF THE DISPERSION INTO ITS COMPONENT PARTS

The following products are likely to be present, viz.: water, ammonia, various chemicals, bituminous matter, animal and vegetable oils or fats, other forms of non-bituminous organic matter and mineral matter.

Test 98. Distillation Residue. The following method has been standardized:⁸

Method (I): The following apparatus will be required:

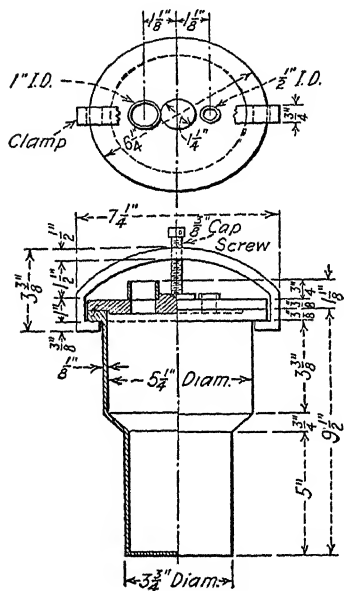
(a) **Iron Still:** The iron still shall be approximately 6 by 3½ in. in inside diameter and adjustable ring burner with holes on the inner periphery to fit around the outside of the still.

NOTE.—A modification of this still is shown in Fig. 382. It consists of the regular still with an expansion chamber superimposed thereon. Two additional ring burners are required, one approximately 6 in. in inside diameter with the holes bored on the inside periphery, and one approximately 2 in. in diameter with the holes bored on top.

(b) Connecting Apparatus: A connecting tube, tin shield, condenser trough, condenser tube and graduated cylinder, as shown in Fig. 383.

(c) Thermometer: A thermometer graduated from 30 to 580° F. (0 to 300° C.).

NOTE.—The details of the assembly of apparatus for the distillation test are illustrated in Fig. 383.



Courtesy A.S.T.M.

FIG. 382.—Iron Still for Use with Badly Foaming Emulsions.

Exactly 200 g. of a well-mixed and representative sample of the emulsion shall be placed in the previously weighed iron still (including lid, clamp, thermometer and gasket, if gasket is used). A gasket of oiled paper may be used between the still and its cover or the joint ground to a tight fit. The cover shall be clamped securely on the still. The thermometer shall be inserted through the small hole in the cover, using a cork stopper, so that the end of the bulb is $\frac{1}{4}$ in. from the bottom of the still. The ring burner shall be placed around the still and the heat applied by this means to the top of the still. Just enough heat from a Bunsen burner shall also be applied to the connecting tube to prevent condensation of water in this tube.

After practically all the condensate has been removed from the still and the temperature of the residue has reached 250° F. (121° C.), the position of the heat from the ring burner shall be lowered to midway of the still and held there until the thermometer reaches 349° F. (176° C.). The burner shall then be rapidly lowered to within $\frac{1}{4}$ in. of the bottom of the still and the temperature increased to and maintained at 500° F. (260° C.) for 15 min. This latter period of heating is necessary to insure a smooth homogeneous residue in the still. At the expiration of the heating period at the maximum temperature, the still and accessories shall again be

weighed and the percentage residue calculated and reported. The cover shall then be removed from the still and suitable portions of the residue shall be poured immediately through a No. 50 sieve into suitable molds and containers for making the required tests. The residue in the molds and containers shall be permitted to cool, uncovered, to laboratory room temperature and thereafter tested for specific gravity (Test 7), penetration (Test 9), ductility (Test

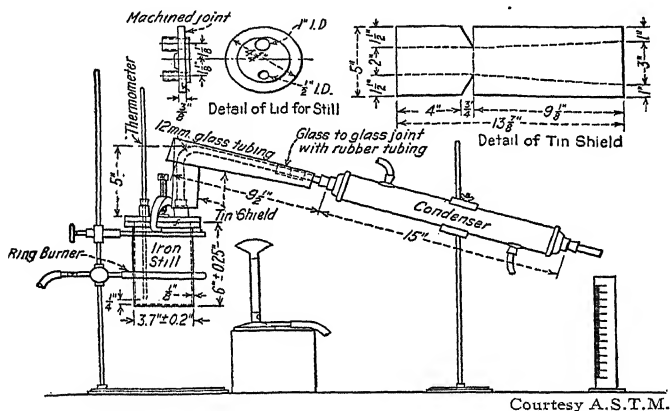


FIG. 383.—Apparatus Assembly for Distillation Test of Emulsified Asphalts.

10), fusing-point (Test 15), solubility in carbon disulfide and ash (Test 21).

It should be noted that the distillation residue will carry any non-volatile dispersing agents present in the emulsion.

NOTE.—When it appears impossible to distil an emulsified asphalt in the still described above due to excessive foaming of the emulsion, then the modified still shown in Fig. 382 should be substituted for the still shown in Fig. 383 and the following procedure followed: Place the 6-in. burner around the larger diameter of the still near its top. This serves as a support. Place the 4-in. burner immediately beneath the flare and the 2-in. burner not less than 2 in. below the bottom. Distillation is started with only the 2-in. burner lighted. Practically all of the distillate should be over in about 45 min. When the distillation apparently stops, light the two larger ring burners and adjust to a low flame. Distillation resumes and when it stops again increase the heat by adjusting the flame of the 2-in. burner. When the temperature can be read upon the thermometer, increase the rate of heating by raising the flame on both the 2- and 4-in. burners and bring the temperature to 500° F. (260° C.). If any evidence is noted of the emulsion beginning to foam over in the delivery tube, remove the 2-in. burner quickly and raise a pan of water so as to immerse the still bottom to a depth

of about 2 in. for a moment, which will check the foaming. Upon resumption of heating watch delivery tube carefully and repeat treatment if necessary.

When the residue has reached and remained at 500° F. (260° C.) for 15 min., proceed as described above for the regular still. While the distillation should be completed in not less than 1 hr. nor more than 1¼ hr. from the first application of heat to the still, the maximum stated is not mandatory as a longer time may be required in some cases to avoid foaming of the emulsion into the condenser. If the residue in the still prior to pouring the ductility and penetration specimens appears granular or heterogeneous in any way, stir with a spatula until the material runs from the spatula in strings instead of drops, and then pour.

Method (II): An alternate method of recovering the asphalt is as follows:¹² Place 200 ml. of the emulsion in an 800-ml. beaker, and in alternate, successive increments, add 50 ml. ethyl alcohol (95 per cent) and 50 ml. anhydrous acetone, and stir until the emulsion has broken. Discard the supernatant layer of alcohol-acetone-water. Then dissolve the residue in 100 ml. ethylene dichloride, boil 20 to 30 min. under a reflux condenser, and while hot, filter through coarse filter-paper into a 500 ml. separatory-funnel. Wash the flask with 25 ml. hot ethylene dichloride to remove all traces of asphalt. Allow the funnel to stand until the layers separate completely, then draw off the lower layer of ethylene dichloride and recover the asphalt as described in Test 21b.

Method (III): Another procedure consists in first freezing the emulsion, and thereupon leaching it with chilled ethyl alcohol (which removes the water), whereupon the asphalt is separated by filtration.¹³

Test 99. Water and Volatile Oils. The following method has been standardized:¹⁴

Method (I): This method of test determines water existing in a sample of bituminous emulsion by distilling the sample with a volatile solvent.

The apparatus shall consist of a metal still or glass flask, heated by suitable means and provided with a reflux condenser discharging into a trap connected to the still or flask. The trap serves to collect and measure the condensed water and to return the solvent to the still. The type of distilling apparatus used is not an essential feature of this method.

(a) *Metal Still*: The metal still, Fig. 331(a), shall be a vertical cylindrical vessel, preferably of copper, having a faced flange

at the top to which the head is tightly attached by means of a clamp. The head shall be of metal, preferably of brass or copper, and be provided with a tubulation 1 in. in inside diameter.

(b) Glass Still: The glass flask, Fig. 331 (b), shall be of the short neck, round-bottom type, made of well-annealed glass, having an approximate capacity of 500 ml.

(c) Heat Source: The burner used with the metal still shall be a ring gas burner 100 mm. (4 in.) in inside diameter. With the glass flask, an ordinary gas burner or electric heater may be used as the source of heat.

(d) Condenser: The condenser shall be of the water-cooled, reflux, glass-tube type, having a condenser jacket not less than 400 mm. (15¾ in.) in length with an inner tube 9.5 to 12.7 mm. (⅜ to ½ in.) in outside diameter. The end of the condenser to be inserted in the trap shall be ground off at an angle of 30 ± 5 deg. from the vertical axis of the condenser.

(e) Trap: The trap shall be made of well-annealed glass constructed in accordance with Fig. 384 and shall be graduated from 0 to 25 ml. in 0.1-ml. divisions. The tolerance of the graduations between 0 and 2 ml. shall be ± 0.5 ml. and between 2 and 25 ml. shall be ± 0.1 ml. The outside diameters should be preferably 2.5 to 3.5 mm. (⅜ to ⅙ in.) greater than the inside diameters specified.

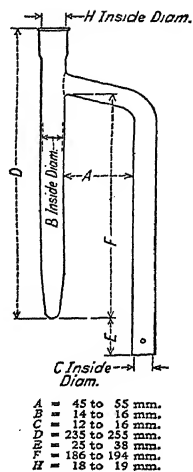


FIG. 384.—Apparatus for Determining Water.

The solvent used when testing bituminous emulsions shall be a coal-tar naphtha or a light oil and shall conform to the following distillation requirements (A.S.T.M. Designation: D 86) :

98 per cent shall distil between 248° F. (120° C.) and 482° F. (250° C.).

The sample shall be thoroughly representative of the material to be tested and the portion of the sample used for the test shall be thoroughly representative of the sample itself. Deviation from this requirement shall not be permitted.

NOTE.—The difficulties in obtaining proper representative samples for this determination are unusually great, so that the importance of sampling cannot be too strongly emphasized.

When the material to be tested contains less than 25 per cent of water, exactly a 100-g. sample shall be placed in the still or flask.

When the material contains more than 25 per cent of water, the sample shall be 50 g. The sample to be tested shall be thoroughly mixed with an equal volume of solvent by swirling, proper care being taken to avoid any loss of material.

The connections between the still or flask, trap and condenser shall be made by means of tight-fitting corks as shown in Fig. 331 (*a*) and (*b*). The end of the condenser inserted in the trap shall be adjusted to that position which will allow the end to be submerged to a depth of not more than 1 mm. (0.04 in.) below the surface of the liquid in the trap after distillation conditions have been established. When the metal still is used, a heavy paper gasket moistened with the solvent shall be inserted between the lid and flange before attaching the clamp. A loose cotton plug shall be inserted in the top of the condenser tube to prevent condensation of atmospheric moisture in the condenser tube.

Heat shall then be applied and so regulated that the condensed distillate falls from the end of the condenser at the rate of from 2 to 5 drops per sec. The ring burner used with the metal still shall be placed about 3 in. above the bottom of the still at the beginning of the distillation and gradually lowered as the distillation proceeds. The distillation shall be continued at the specified rate until no water is visible on any part of the apparatus except at the bottom of the trap. This operation usually requires less than 1 hr. A persistent ring of condensed water in the condenser tube shall be removed by increasing the rate of distillation for a few minutes.

The volume of condensed water measured in the trap at room temperature multiplied by 100 and divided by the weight of sample used, shall be the percentage of water and shall be reported as "..... per cent water by weight, A.S.T.M. method."

The accuracy to be expected with this method is that duplicate determinations of water should not differ from each other by more than one division on the trap.

The sum of the percentages of Distillation Residue and Water, deducted from 100, represents the percentage of Volatile Oils.

Method (II): A rapid method for determining the amount of water present consists in weighing 50 g. of the well-mixed material into a standard 3-oz. tin box, which is then heated in an oven at 163° C. (325° F.) for five hours, and the loss in weight deter-

mined. Another rapid method consists in precipitating the bituminous constituents from the emulsion with hydrochloric acid, neutralizing with ammonia, and then pressing out the water mechanically, finally weighing the residue. Still another rapid method¹⁵ for separating the water and soluble dispersing agents from the bituminous constituents and insoluble dispersing agents, consists in pouring 15 g. of the well-mixed emulsion onto a porous clay plate, which is then exposed to air at room temperature for 48 hours. The film is then scraped off, weighed, and tested for fusing-point, etc. It is claimed that in this procedure, the composition of the material is not altered in any respect.

Test 100. Dispersing Agents. These may be ascertained as follows:¹⁶ Soaps are determined by adding 25 ml. of 96 per cent ethyl alcohol gradually to a 10-g. sample of the emulsion, and washing the separated residue with 10 ml. alcohol. Small quantities of the bituminous constituents dissolved in the combined alcoholic extracts are removed by adding 20 ml. of water and then shaking with benzol in a separatory funnel. The separated bituminous constituents and benzol extract are then evaporated together and weighed. The bituminous residue is then tested for tar products. The alcoholic extracts are evaporated dry and the soaps are extracted with hot water and transposed with HCl. They may be tested for resin-, naphthenic- and fatty-acids as follows: resin acids are detected by the usual color reaction; naphthenic acids react with formaldehyde and sulfuric acid, forming an ether-insoluble formolite; whereas fatty-acids yield an ether-soluble formolite. The copper salts of naphthenic acids dissolve in benzol, forming a green-colored solution. Ammonia soaps may be detected by adding NaOH and heating. If present, ammonia may be determined quantitatively by distilling a weighed quantity of the emulsion into a standard solution of sulfuric acid and titrating with alkali. Sulfite liquor is extracted from the original emulsion with an equal volume of 70 per cent ethyl alcohol, whereupon the residue obtained on evaporation yields lignosulfonic acid with dilute HCl (which evolves SO_2 on heating), contains calcium, reduces Fehling's solution, and gives the carbohydrate reaction with α -naphthol and sulfuric acid. Inorganic dispersing agents may generally be determined by boiling the emulsion with dilute hydrochloric acid and extracting the bitumi-

nous substances with benzol. The inorganic chemicals will remain in the aqueous layer and may be determined by a qualitative or quantitative analysis. In other cases inorganic agents, such as clay, may be ascertained by incinerating a weighed quantity of the emulsion, and examining the ash. Special methods must be used for determining albumenoids, proteins, gums, alginates, alkaline bases, tannins, polysaccharides, sulfonated oils, etc.

Methods have likewise been described for the analysis of wax-emulsions and oil-emulsions.¹⁷

CHAPTER XXXVII

WEATHERING TESTS

Effects of Weathering. All substances undergo a change on being exposed to air, moisture and sunlight. Metals undergo corrosion, rocks disintegrate, wood decays and animal or vegetable fibers decompose by hydrolysis. Bituminous substances are not immune from such action. On exposure to the weather (i.e., air, sunlight and moisture) they will change physically and chemically.

In the early days of photography, solutions of asphalt in ethereal oils such as turpentine, oil of lavender, etc., were used for preparing the sensitized photographic film. On exposure to light under the lens of a camera, certain changes took place in the asphaltic coating, as evidenced by the fact that upon subjecting it to the action of certain solvents, those portions which had been in contact with light became insoluble, whereas those protected from its action readily dissolved in the solvent, bringing the photographic image into relief. It took rather a long exposure to produce satisfactory images, since asphalt is only moderately sensitive to light, in comparison with some of the high-speed photographic plates in use at the present time. Nevertheless very artistic results have been produced by this crude method, which was originally discovered in 1816 by Joseph N. Nicpce, who used a solution of asphalt in "Dippel's oil" obtained as a distillate in the destructive distillation of bones. In other experiments, he used oil of lavender and petroleum distillates to remove the asphalt unaffected by the light, followed by a final washing with warm water.¹

It was soon observed that certain forms of asphalt were more photo-sensitive than others, and Syrian asphalt in particular became very popular on account of its purity, solubility, hardness and sensitiveness to the light's rays.² It was subsequently found that the addition of sulfur chloride increased the sensitiveness of native asphalts,³ but petroleum asphalts were apparently rendered inert in its presence. Further investigations revealed the fact that petro-

leum asphalts free from paraffin are relatively the most sensitive towards light,⁴ likewise mixtures of asphalt and rubber or gutta percha.⁵

Maximilian Toch noted that bituminous materials on exposure to sunlight decomposed with the liberation of "free carbon." ⁶ His experiments indicated that this action was inhibited by incorporating an opaque pigment. He pointed out further, that animal and vegetable oils (triglycerides) are not affected in this manner, and when blended with bituminous materials, apparently retard the action. Archaeological excavations indicate that asphalt coatings undergo no appreciable deterioration during thousands of years in the dark, but on exposure to light, rapid surface changes occur.⁷

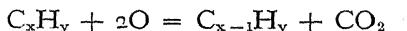
Investigations of the weathering of bituminous substances have been conducted by Hubbard and Reeve,⁸ Church and Weiss,⁹ Reeve and Anderton,¹⁰ also Reeve and Lewis.¹¹ The changes brought about upon exposure to the elements are quite complicated, involving one or more of the following reactions:¹²

Evaporation. This represents the gradual loss of volatile constituents on exposure to air and the sun's heat. Certain bituminous materials evaporate quite rapidly, and especially the tars.¹³ With any bituminous substance, the rate of evaporation depends almost entirely upon the temperature. Other things being equal, the higher the temperature the greater will be the volatilization. The determination of volatile matter (Test 16) is usually regarded to be an accelerated evaporation test, which is supposed to show in a relatively short time at an elevated temperature, what takes place over a lengthy period when exposed naturally to the air and sun. This is not, however, strictly correct, as will be explained below.

Oxidation. This takes place on exposure to air and progresses more rapidly at high than at low temperatures. The effect of oxidation is two-fold, and involves the direct union of oxygen with the bituminous substances, also the elimination of a portion of the hydrogen or carbon in the form of water or CO₂.¹⁴ These reactions may be expressed roughly as follows:



also

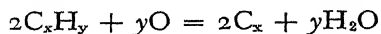


The absorption of oxygen is accompanied by a *gain* in weight whereas the elimination of hydrogen or carbon is accompanied by a *loss* in weight. At low temperatures, these reactions are probably induced to a large extent by the actinic light rays.

A method has been proposed¹⁵ for quantitatively measuring the volume of oxygen absorbed upon heating asphalts at 200° C. for 3 hours, the resultant changes in physical characteristics and components being recorded. One gram of the asphalt is intimately mixed with 25 g. of standard A.S.T.M. silica sand in a 50-ml. Erlenmeyer flask, which is heated in an oil-bath maintained at 200° C., while a flow of oxygen passes over the sample under a constant pressure of 2–3 mm. of xylene. The volume of oxygen absorbed is measured in ml. during 3 hours' heating. The water and CO₂ evolved are determined, likewise the loss or gain in weight of the sample. By this method, it has been found that the asphaltenes gain in weight during the reaction, whereas the asphaltic resins, petroleum resins and oils lose in weight. Moreover, asphaltenes from gilsonite and petroleum asphalts derived from U. S. petroleum, oxidize less than the asphaltenes separated from Mexican petroleum asphalt.

It is recognized that bituminous substances behave differently when heated in an inert atmosphere such as illuminating gas or nitrogen, than when heated under similar conditions in air or oxygen. In the former instance evaporation only takes place, whereas in the latter, evaporation occurs as before, but this at the same time is accompanied by a loss in weight due to elimination of hydrogen, also by a gain in weight caused by the absorption of oxygen. The extent and nature of these reactions will depend upon the substance itself, and also on the conditions to which it is subjected.

Carbonization. This represents the formation of "free carbon" in the bituminous material, and is induced by an extensive elimination of hydrogen, as indicated by the following reaction:



In other words, it represents the elimination of hydrogen, carried to an extreme. As a matter of fact, the deposit of free carbon generally contains a small percentage of hydrogen, and is rarely composed of pure carbon. This reaction progresses most rapidly

in sunlight, but will similarly take place upon subjecting the bituminous substance to a high temperature (see "Overheating").

Polymerization. This is due to a condensation or polymerization of the molecules, and manifests itself by a hardening or "setting" of the substance.¹⁰ This polymerization has also been termed "thixotropy" or "spontaneous hardening" and is comparable, in a way, to the hardening or setting of portland cement. It occurs usually to a greater extent on the surface of the material than it does beneath. The reaction may be expressed as follows:



Bituminous materials after being freshly melted will appear softer and show a lower fusing-point than upon standing a day or two. For this reason it is recommended that the hardness and fusing-point be determined on the freshly melted material.

A method has been described¹⁷ for quantitatively measuring the increase in viscosity of asphalts with time, by means of the Falling Coaxial Cylinder Method (Test 8*h*), the viscosity being expressed in absolute units (poises) at atmospheric temperatures. By plotting the log-viscosity against the time on a logarithmic scale, straight lines are obtained, the slope of which give quantitative measures of the rates of hardening. Assuming 100 hours as the basis for comparing various asphalts, the following expression results, which is termed the "Asphalt Aging Index":

$$A.A.I. = m$$

where m is the slope of the log-viscosity versus log-time curve. In this manner, it has been ascertained that the hardening is not caused by surface evaporation, and furthermore, that gentle heating of the samples brought their viscosities back almost to the original values.

Polymerization similarly takes place to a greater or lesser extent on heating bituminous materials to a high temperature, and is especially noticeable in fatty-acid pitches, some of which set and become infusible upon being heated in the neighborhood of 300° C., in the same manner as china-wood oil.

The more highly the asphalt is blown, the more rapid will be the increase of its consistency with time. Due to its reversible na-

ture, the age-hardening phenomenon may be considered as a form of thixotropy.

Effects of Moisture. All bituminous substances are more or less affected upon exposure to moisture, which manifests itself in two ways, namely by the actual absorption of water and by the gradual leaching out of soluble constituents. These actions become intensified when the substance has oxidized, since oxygenated substances seem to have a greater affinity for moisture than the hydrocarbons themselves.

The moisture-absorbing properties of bituminous substances may be demonstrated optically, by pasting a postage stamp on a piece of glass and coating it with a film of the bituminous substance applied in the form of paint. After the solvent has evaporated, the sheet of glass is immersed in water. Within twenty-four to forty-eight hours the water will be observed to have permeated the film, loosening the postage stamp, and forming a blister underneath.¹⁸

It has also been noted¹⁹ that the bleaching of natural rock asphalts upon exposure to the weather is occasioned by the oxidation of the asphaltic constituents, which are thereupon readily leached out under the influence of atmospheric moisture. In addition, it has been found²⁰ that samples of asphalt, when exposed to the action of an arc-light, are gradually converted into water-soluble products containing acid- and ketone-bodies. Both light and oxygen are necessary factors for the formation of soluble products. These show an acid reaction and are precipitated by basic lead acetate.

The evaporation of volatile constituents upon aging leaves pores, which serve to increase the moisture-absorption, and at the same time permit free ingress of atmospheric oxygen. The latter serves to oxidize the bituminous constituents into water-soluble products, which in turn are leached out by the water, thereby increasing the porosity and augmenting the process of disintegration. The presence of clay or other associated mineral, animal, or vegetable constituents having an affinity for moisture, serves to hasten the destruction of bituminous substances. Animal and vegetable fibers associated with bituminized fabrics, in contact with moisture, are likewise decomposed by the ravages of mildew and molds.

The following constitutes a résumé of the changes occasioned by the weathering process:

Gains:

- (1) Oxidation products formed through the addition of oxygen.

Losses:

- (2) Evaporation of the volatile constituents.
 (3) Elimination of hydrogen (as H_2O) by oxidation.
 (4) Elimination of carbon (as CO_2) by oxidation.
 (5) Leaching out of soluble oxidation products (in item 1) by the action of water.

Alterations:

- (6) Polymerization.
 (7) Generation of free carbon.

It should be noted that there is more artificial hardening due to causes other than loss of volatile matter in actual weathering, than under the laboratory heat tests. There is no relation between the ductility of the residue in the laboratory heat test and the ductility of the substance in actual weathering. Substances showing carbenes will rapidly undergo carbonization under actual weathering, and the reverse is also true.²¹

Exposure to the weather affects the physical and chemical characteristics of bituminous substances in the following manner, viz.:

(Test 1)	Color in mass.	Becomes lighter
(Test 2)	Homogeneity.	Destroyed by the formation of free carbon
(Test 5)	Lustre.	Disappears, the surface becoming dull
(Test 6)	Streak.	Often changes from a black to a brown, and sometimes to a yellow
(Test 7)	Specific gravity.	Increases
(Test 8)	Viscosity.	Increases
(Test 9)	Hardness.	Increases
(Test 10)	Ductility.	Decreases
(Test 11)	Tensile strength.	Decreases
(Test 12)	Adhesiveness.	Decreases
(Test 13)	Breaking-point.	Increases
(Test 14)	Solidifying-point.	Increases
(Test 15)	Softening-point.	Increases
(Test 15g)	Flow-point.	Increases
(Test 15h)	Liquefying-point.	Increases
(Test 15i)	Twisting-point.	Increases
(Test 16)	Volatile matter.	Decreases
(Test 17)	Flash-point.	Increases
(Test 18)	Burning-point.	Increases
(Test 19)	Fixed carbon.	Increases
(Test 21)	Solubility in carbon disulfide.	Decreases
	Non-mineral matter insoluble.	Increases
(Test 22)	Carbenes.	Variable
(Test 23)	Solubility in 88° petroleum naphtha.	Decreases
(Test 24)	Free carbon.	Increases
(Test 37f)	Unsaponifiable constituents.	Unchanged

(Test 37g)	Saponifiable constituents.....	Unchanged
(Test 37h)	Glycerol.....	Unchanged
(Test 38c)	Asphaltenes.....	Increase
(Test 38d)	Asphaltic resins.....	Unchanged
(Test 38e)	Oily constituents.....	Decrease

The weather-resisting properties of bituminous substances are of primary importance in the case of bituminized roof coverings, bituminous lacquers, cements, varnishes and enamels, on account of the relatively *thin* layers in which these products are customarily employed.

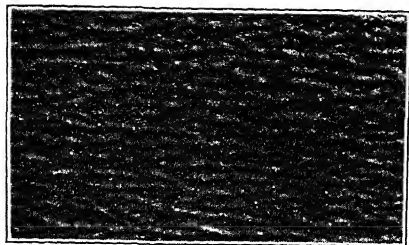
Two classes of weathering tests may be used, one of which consists in actually exposing the material to the effects of the elements, and the other in subjecting the material in the laboratory to the influence of artificial conditions of light, moisture and heat which duplicate as closely as possible the natural agencies, but in an intensified form. The latter has been aptly termed an "accelerated" weathering test.

Test 101. Actual Weathering Test. This test is usually slow, but the results are unquestionably reliable, since they duplicate actual service conditions.

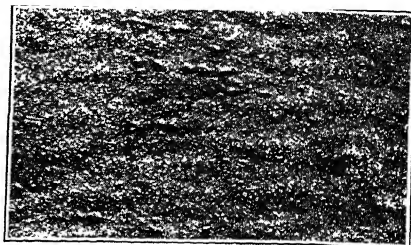
(1) *Testing Bituminized Fabrics:* The following system has been adopted by the author for conducting exposure tests on bituminized roll-roofings, viz.: Sections 18 in. by 36 in., or 18 in. by 32 in., depending upon whether the roofing is 36 or 32 in. wide, are taken across the sheet, the cutting being sharp and square. These are exposed on a platform, composed of $\frac{7}{8}$ -in. tongued and grooved boards, preferably pine, having a 2-in. slope to the south, the samples being nailed with large-headed galvanized barbed roofing nails at the four corners, midway across the 18-in. edges, and at three intermediate points along the 26- or 32-in. edges, a total of 12 nails being used.

Both the indoor and exposed samples are examined at the following intervals, viz.: one-half year, one, two, three, four, five and ten years, and the data recorded relative to the appearance and condition of the specimens.

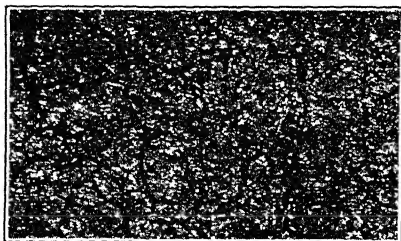
The eight specimens illustrated in Fig. 385 represent the typical surface conditions of smooth-surfaced roll-roofings after exposure to the weather for a period of 5 years. Specimen *A* is unchanged and homogeneous, specimen *B* is covered with fine checks, specimen



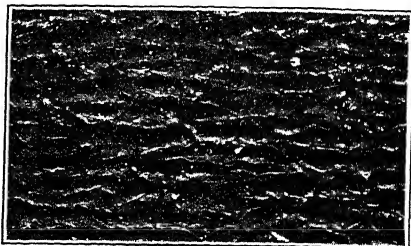
(A)



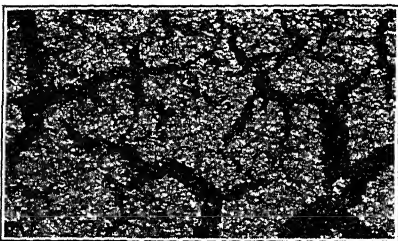
(E)



(B)



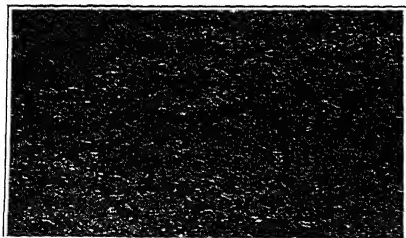
(F)



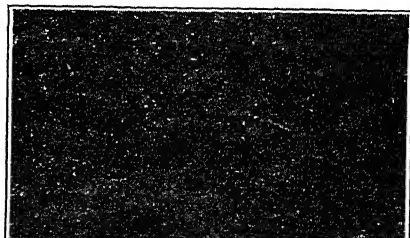
(C)



(G)



(D)



(H)

FIG. 385.—Effects of Exposure on Smooth-surfaced Prepared Roofings.

C is covered with coarse checks, in specimen *D* the checks are disappearing (i.e., they were in evidence at the time of the previous observation, but have since largely disappeared), specimen *E* shows a few blisters, specimen *F* is covered with hair cracks, specimen *G* is covered with coarse cracks, and in specimen *H* the felt is largely exposed. Figure 386 shows specimens *A* and *H* enlarged $3\frac{1}{2}$ diameters. The veined surface of *A* shows up very distinctly, and also

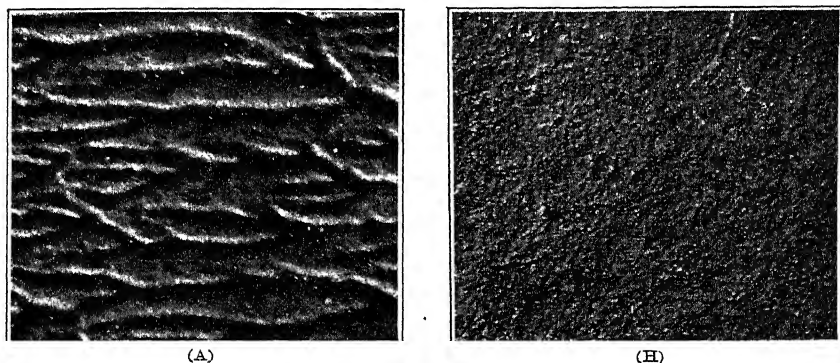


FIG. 386.—Enlargements of Specimens A and H in Fig. 385.

the characteristic uneven appearance of the roofing when the weather-coating has worn off and the felt fibers exposed, as in *H*.

“Checking” is distinctly a surface phenomenon which manifests itself with certain substances on exposure. The checks rarely extend entirely through the bituminous coating, and are seemingly caused by the hardening and contraction of the upper stratum (i.e., shrinkage of the skin), resulting in a tension which is sufficient to cause it to crack and slide over the softer sub-stratum. One theory is that checking or “grooving” is caused by the difference in surface-tension between the oily constituents and the asphaltenes, under the influence of light. Bituminous substances which are largely influenced by changes in temperature (in other words having a high susceptibility index) are likely to check. As the “spontaneous hardening” progresses downward into the lower layers, the checks gradually disappear. It has been suggested that the contraction and checking of asphalts may be obviated by adding up to 10 per

cent by weight of coal-tar pitch, or 2 to 3 per cent by weight of anti-oxidants, such as are used in the rubber industry. Antioxidants are more correctly termed "antioxygens." There are three general classes: (1) amines, (2) phenols, and (3) aldehyde-amine condensation products. The antioxygens that have been proposed for use with bituminous substances include: phenyl-beta-naphthylamine, phenyl-alpha-naphthylamine, hydroquinone, cachetol, pyrocachetol, guaiacol, dipentene, pyridine, quinoline, aniline, dimethylaniline,²² alkaloids (e.g., nicotine),²³ phenol-formaldehyde resin,²⁴ titanium phthalate,²⁵ nitrogenous bases (e.g., bone tar),²⁶ a mixture of fatty acid and sodium lauryl sulfate,²⁷ amino-mercapto-benzothiazole, tetramethyl-diamino-diphenyl-methane, phosphorus, P_2S_5 , P_4S_6 , etc. The resistance of blown asphalts to actinic rays of light may be improved by incorporating a small percentage of oleic acid, montanic acid, or an oxidized paraffin wax.²⁸

The incorporation of finely-divided fillers will tend: (a) to prevent checking and cracking; (b) to improve the weather-resistance, by protecting the coating from the disintegrative action of sunlight; (c) to give the coating "body," so that it will not flow so readily; (d) to increase its "plastic range" (i.e., by increasing its fusing-point and at the same time decreasing its solidifying-point); and (e) to improve its resistance to mechanical attrition. The optimum characteristics of such fillers are: (a) they shall be finely-divided; (b) they shall be light-excluding; (c) they shall be of themselves chemically inert and resistant to weathering influences (i.e., heat, moisture, acids, etc.); (d) the mixing with the bituminous constituents shall be thorough and complete, so that the mixture will be homogeneous; (e) the filler must be used in suitable proportions; and (f) the use of two fillers will often give better results than the use of either component alone in the same total quantity. Different fillers act differently with any one asphalt. Similarly, each asphalt behaves differently with any particular filler. Hence, every asphalt-filler combination must be tested out individually, to arrive at its suitability. The properties of combinations involving the use of two or more fillers are not always additive. The fusing-point of asphalts is increased by the addition of fillers, and the finer the subdivision of the filler, the greater will be the increase. When a coarse and a fine filler are added together, the

fusing-point of the mixture will be increased more than would be the case if the total quantity of fine filler alone were added.

"Blistering" is caused by one or more of the following factors, viz.:

1. Temperature: If the saturated sheet is at too high a temperature (i.e., above 375° F. in the case of asphaltic saturants) when the coating is applied, or if the coating itself is applied too hot, then non-condensable gaseous vapors are apt to be imprisoned in the bituminous constituents, which under the heat of the sun will expand and form unsightly blisters.

2. Entrapped air: This is one of the principal causes of blistering.²⁹ The air may be entrapped in the coating, because of the prior blowing process, or as a result of incorporating fillers, or in the mechanical operation of applying the melted coating to the sheet of saturated felt. Blistering may also be caused by voids in the saturated felt base, in which event the entrained air or absorbed moisture will expand at elevated temperatures.³⁰

3. Volatile matter: Saturants, and to a lesser extent coatings, which carry an abnormally large proportion of volatile constituents are likely to blister, since these have a tendency to vaporize when subjected to the sun's heat and the resulting pressure can only be relieved in this manner.³¹

4. Fusing-point of the coating: Coatings having high fusing-points are less apt to blister than those made of a low fusing-point, other things being equal. The inclusion of a certain amount of suitable filler in low fusing-point coatings will also tend to overcome this manifestation.

5. Moisture: The absorption of moisture by the sheet, which is rapidly vaporized and expanded by the heat of the sun. In the case of mineral-surfaced roofings and shingles, the porosity of the slate or other mineral surfacing proves a contributing factor, due to the likelihood of porous minerals absorbing moisture and conducting it to the interior of the sheet. Moisture imprisoned in this manner will suddenly expand under the heat of the sun, and by following the path of least resistance, will distort the coating and form pockets or blisters.

"Hair cracks" are caused by the contraction of the bituminous material, and take place with substances which are hard, brittle,

and devoid of elastic properties. The action is aggravated by the use of soft, plastic saturating materials in conjunction with a hard and brittle coating. The cracks usually extend all the way through the bituminous coating, and will neither seal up nor disappear in time, as is the case with the checks.

"Bleeding" or exudation of the saturant, with the resulting formation of dark-colored spots or blotches over the surface of the sheet, causing an unsightly appearance, is claimed to result from the formation of a thin layer of asphalt between the saturant and the coating, of a softer consistency than either, which after a period of time migrates through the coating by capillarity.³²

"Predominating color" is a criterion of the rapidity with which the soapstone or mineral matter on the surface disappears, and "dusting" furnishes an indication of the rate with which the bituminous coating weathers away on exposure. As bituminous substances weather, they form a pulverulent chalk-like mass having but little coherence, and which is therefore easily removed by wiping with a cloth. This corresponds to "chalking" of linseed-oil paint films. The influence of rubbing on the color is of supplemental value, furnishing an indication of how deep the weathering has progressed.

"Pliability" of the roofing shows to what extent the roofing has "dried out," bearing in mind that when the pliability decreases to a certain extent, the sheet can no longer fulfil its function properly, but will break upon being subjected to severe vibratory strains.

"Tensile strength" indicates the extent to which the weathering has weakened the roofing, also an approximation of its residual wearing qualities. By constructing a curve of the tensile strength of the sheet at different periods, some idea may be gained of its probable durability. As the roofing ages indoors, it gradually gains in strength, until it finally remains constant. A duplicate specimen exposed outdoors rapidly gains in strength up to a certain point, which corresponds to the disappearance of its weather coating. The tensile strength will thereupon decrease until it falls below the corresponding strength of the indoor sample. The roofing reaches its "mean effective life" when the strength curve of the outdoor sample crosses the curve of the indoor sample. This will be made clear by Fig. 387, showing the strength curves of repre-

sentative high-grade light, medium and heavy weight smooth-surfaced prepared roofings, weighing 32, 42 and 52 lb. net per 108 sq. ft. The solid lines represent the strength of the outdoor samples, and the dotted lines the corresponding strength of samples aged indoors. The figures in Table CLVII will interpret the diagram.

The mean effective lives of the roofings in question may be taken as nine, twelve and one-half and fifteen years respectively, and the maximum effective lives as ten, thirteen and fifteen and one-half years.³³

(II) *Testing Bituminous-solvent Compositions*: These may similarly be tested by applying them in one or more coats to steel sheets or wooden panels, and observing their appearance at regular intervals. The following features should be recorded:³⁴

- (1) Loss of lustre.
- (2) Condition of the exposed surface.

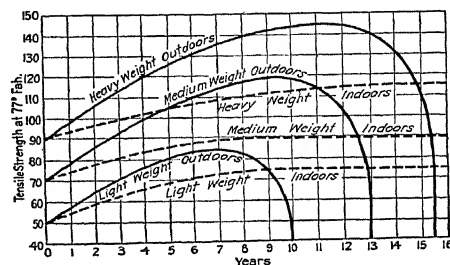


FIG. 387.—Tensile Strength Curves of Prepared Roofings on Exposure.

TABLE CLVII

EFFECT OF ACTUAL WEATHERING ON THE STRENGTH OF PREPARED-ROOFING

	Light Weight (35 lb. per 100 sq. ft.)		Medium Weight (45 lb. per 100 sq. ft.)		Heavy Weight (55 lb. per 100 sq. ft.)	
	Years	Lbs.	Years	Lbs.	Years	Lbs.
Original strength.....	0	50	0	70	0	90
Maximum strength outdoor sample.	7	85	10	120	12	145
Outdoor sample same strength as indoor. .	9	75	12½	90	15	115
Outdoor sample same strength as originally	10	50	13	70	15½	90

- (3) Amount of dusting.
- (4) Influence of rubbing on the color.
- (5) Any chipping of the composition and exposure of the underlying surface.
- (6) Any corrosion in the case of the steel plates.

Much has been written concerning the methods for performing exposure tests on rubber, paints, including bituminous coatings and dispersions, and for further information on this subject, the reader is referred elsewhere.³⁵

(III) *Testing Crude, Refined or Blended Bituminous Substances*: This test is performed by melting the bituminous substance at the lowest possible temperature in a shallow, flat-bottomed glass dish in a layer 0.025 in. thick. A so-called petri dish may conveniently be used for this purpose, measuring approximately 3 in. in diameter and $\frac{1}{2}$ in. deep, thereby exposing a surface of approximately 7 sq. in. (451.6 sq. mm.). This will require 3 ml. of the material, which, multiplied by its specific gravity, will give the corresponding weight in grams. The proper weight of bituminous substance is introduced into the dish and then melted in an air-bath at a temperature slightly above its fusing-point, so as not to result in any appreciable loss of volatile constituents. An alternate procedure consists in applying a layer of the bituminous material 0.025 in. thick to a sheet of thin aluminum, 3 by 6 in., and 0.050 to 0.065 in. thick. This may be conveniently done by melting the material in a layer somewhat thicker, then placing the specimen in a mold of the proper depth (i.e., the thickness of the metal sheet plus 0.025 in.), and finally rolling it cold between a pair of heated metal rollers until all but 0.025 in. \pm 0.003 in. of the bituminous coating is removed. The dishes or panels are exposed to the elements in a dustproof box having a quartz glass top, so as to permit access of the largest amount of ultra-violet rays. Air circulation is provided by means of side vents covered with cotton gauze and the specimens are protected from rain by overhanging eaves. The specimens are examined at the end of six months and one year, and the following observations recorded:

- (1) Appearance to the eye and under the microscope.
- (2) Amount of dusting and influence of rubbing on the color.
- (3) Increase in softening-point over that of the original material. (Test 15.)
- (4) Comparison of the breaking-point (Test 13) with that of the original material (where the test is made on sheet metal).
- (5) Increase of the amount of material insoluble in carbon disulfide over that originally present. (Test 21.)

The effect of weathering upon the ductility may be ascertained by coating strips of rubber with a layer of the bituminous substance

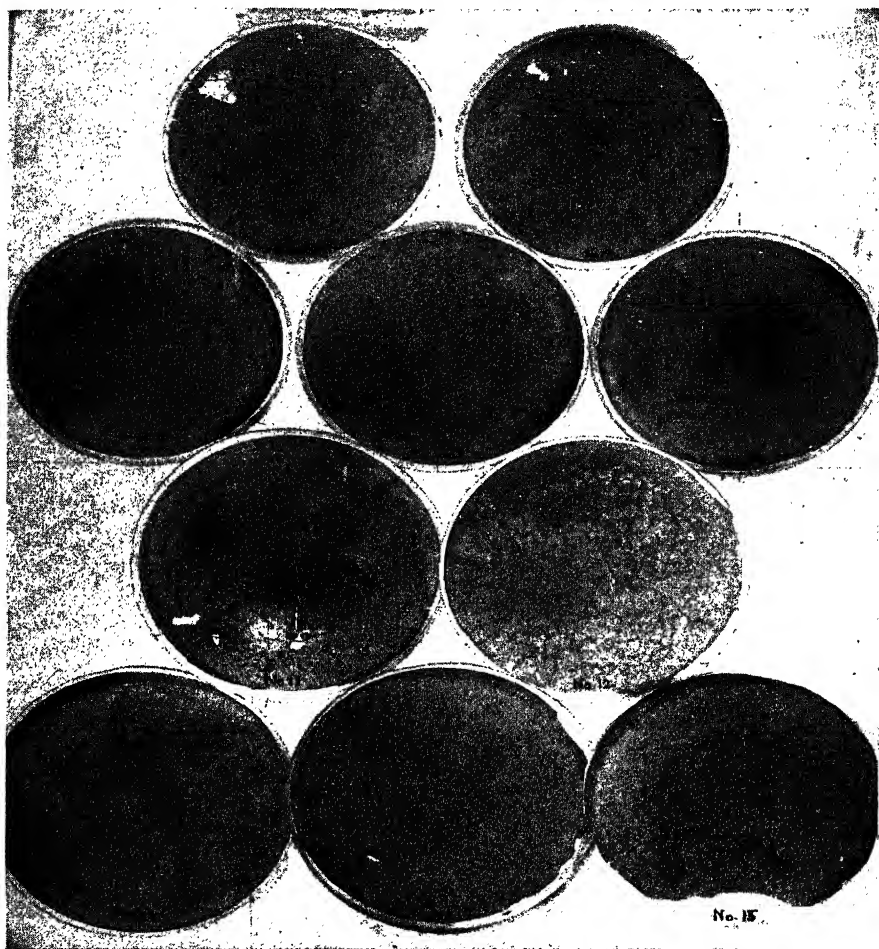


FIG. 388.—Typical Bituminous Substances after One Year's Exposure to the Weather.

0.005-in. \pm 10 per cent in thickness, and noting the critical temperature at which the film cracks when elongated 33 per cent under prescribed testing conditions. Duplicate determinations are run

both before and after exposure to the weather, or to the accelerated test.³⁶

Figure 388 illustrates the appearance of typical specimens after having been exposed for one year. In the case of asphalts, the fusing-point increased up to 150° F. over the original fusing-points, and the material insoluble in carbon disulfide showed an increase ranging from 1 to 10 per cent over the original figures.

Tests have been conducted³⁷ to ascertain the effects of weathering on the asphaltic constituents separated in accordance with Test 38. The results obtained from Mexican, German and Russian residual asphalts are given in Table CLVIII. It will be observed that the oily constituents progressively decrease, whereas the as-

TABLE CLVIII

EFFECT OF ACTUAL WEATHERING ON THE CHARACTERISTICS OF PETROLEUM ASPHALTS

	Mexican Residual Asphalt	German Residual Asphalt	Russian Residual Asphalt
<i>Original Material:</i>			
Fusing-point (K. and S.).....	24.8° C.	28.1° C.	30.7° C.
Dropping-point (Ubbelohde).....	50.8° C.	50.3° C.	50.8° C.
Oily constituents.....	49.29%	39.11%	46.67%
Asphalt resins.....	24.76%	43.75%	32.17%
Resins in asphaltenes.....	21.34%	16.02%	20.55%
Hard asphalt in asphaltenes.....	4.61%	1.12%	0.61%
Total.....	100.00%	100.00%	100.00%
<i>After Exposure in Dark to Air Indoors 5 Mos. (layer $\frac{1}{4}$-$\frac{1}{2}$ mm.):</i>			
Fusing-point (K. and S.).....	+ 6.4° C.	+ 3.3° C.	+ 0.1° C.
Dropping-point (Ubbelohde).....	+ 3.2° C.	+ 4.7° C.	+ 1.2° C.
Oily constituents.....	- 9.33%	- 2.39%	- 1.03%
Asphalt resins.....	+14.77%	- 0.50%	+ 1.51%
Resins in asphaltenes.....	- 5.56%	+ 2.66%	- 0.62%
Hard asphalt in asphaltenes.....	+ 0.12%	+ 0.23%	+ 0.14%
<i>After Exposure to Weather Outdoors 5 Mos. (layer $\frac{1}{4}$-$\frac{1}{2}$ mm.):</i>			
Fusing-point (K. and S.).....	+41.4° C.	+29.3° C.	+25.2° C.
Dropping-point (Ubbelohde).....	+39.5° C.	+29.7° C.	+26.7° C.
Oily constituents.....	-20.75%	-13.59%	- 8.43%
Asphalt resins.....	+ 5.89%	- 8.57%	- 9.42%
Resins in asphaltenes.....	+13.11%	+18.40%	+13.03%
Hard asphalt in asphaltenes.....	+ 1.75%	+ 3.76%	+ 4.82%

phaltic resins and the hard asphalt in the asphaltenes are correspondingly increased.

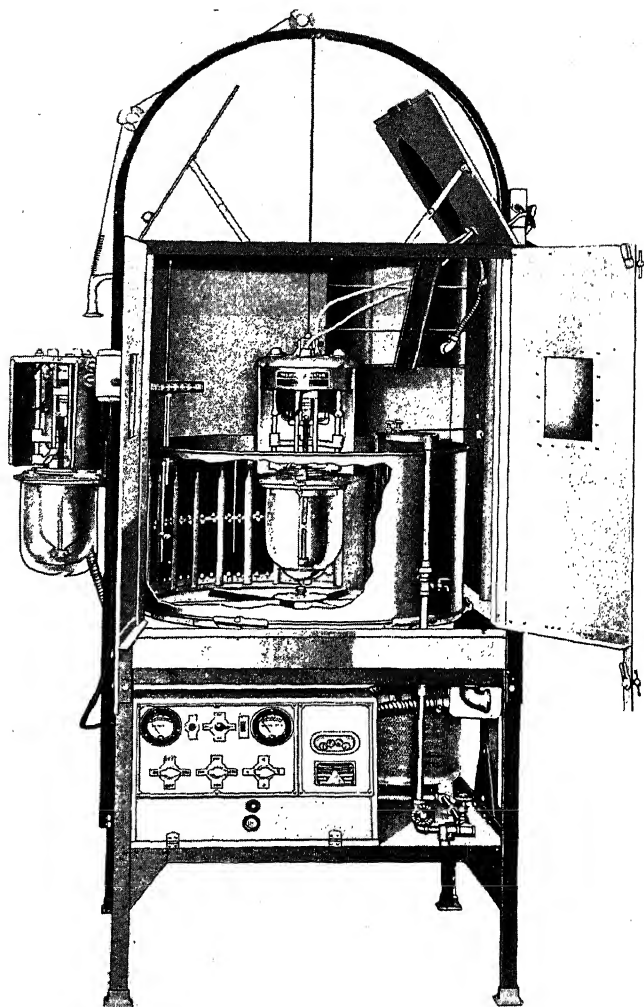
Test 101a. Testing Thin Films. Tests have also been described,³⁸ including a method of oxidizing thin films by heating to 140° F. in admixture with potassium permanganate acidified with sulfuric acid, and thereupon ascertaining the consumption of the potassium permanganate by titration.³⁹ Tests have also been suggested to evaluate road tars by "accelerated" procedures of one kind or another.⁴⁰ An example consists in mixing 98 per cent by weight of 20- to 30-mesh Ottawa silica sand with 2 ± 0.05 per cent by weight of the tar, and packing 100-g. portions of the mixture into aluminum tubes 9 in. long by 1 in. in diameter (held in place on the bottom by a cloth gauze and on top by a steel-wool plug). Filtered and oil-free air maintained at $30 \pm 0.1^\circ$ C. is then passed through the tubes at a rate of 50 to 60 cu. ft. per hr. Weighings before and after the elapse of 23 hrs. give the loss by evaporation, which is calculated in percentage of the tar present in the mixture (having tar films present in the order of 0.006 mm. in thickness). This test is claimed to furnish an indication of the weathering characteristics of the tar.⁴¹

Test 102. Accelerated Weathering Test. The apparatus developed for this purpose has been developed by the Bureau of Standards, Washington, D. C.⁴² The following procedure has been proposed for testing bituminous substances:

This method is intended to determine the durability of bituminous materials by producing rapid deterioration of the materials under conditions simulating extreme outdoor exposure.

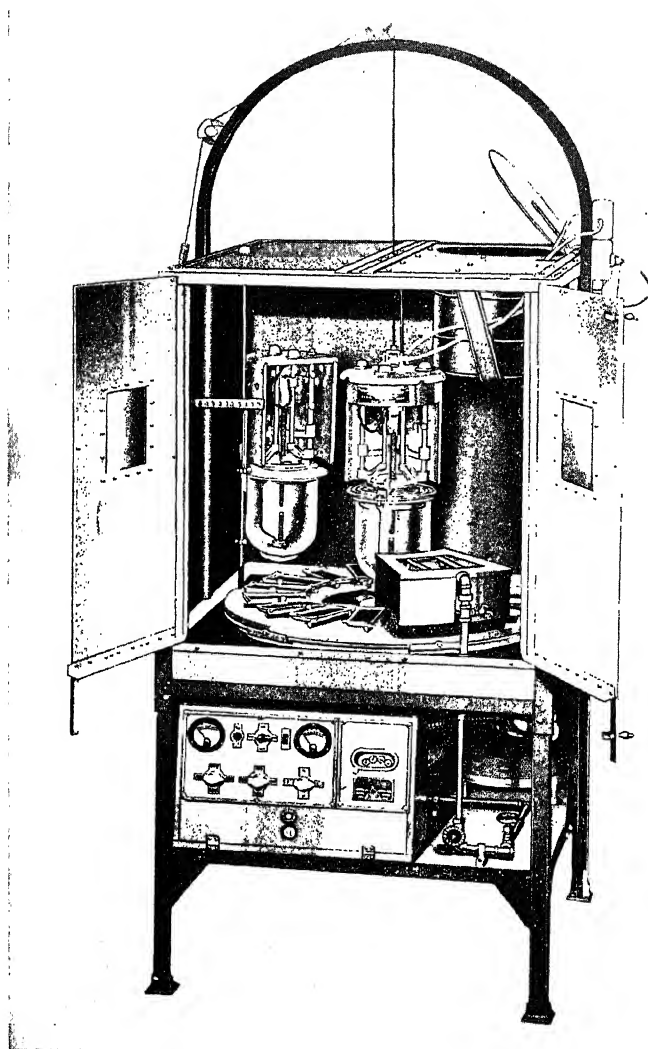
The apparatus required consists of the actual weathering equipment, a trimmer to prepare the bituminous coatings and an instrument to determine the extent of the "weathering," as follows:

Accelerated Weathering Equipment: The actual weathering equipment shall consist of a carbon-arc lamp, a cylinder, a sprinkler, a spray, and an arrangement for temperature control during the light period. Fig. 389 shows a complete unit suitable for testing the materials in the vertical position. Soft materials are tested in the horizontal position, using one or two lamps as shown in Fig. 390.



Courtesy Bureau of Standards.

FIG. 389.—Accelerated Weathering Apparatus for Testing Panels in the Vertical Position.



Courtesy Bureau of Standards

FIG. 390.—Accelerated Weathering Apparatus for Testing Soft Materials in the Horizontal Position.

Carbon-arc Lamps: The following specifications have been standardized:⁴³

This recommended practice covers the basic principles of an accelerated weathering unit of the carbon arc type.

NOTE.—Inasmuch as weather varies from day to day and place to place, thus making it impossible to correlate with the weather at all times and for all locations, it is desirable to promote the adoption of such cycles as will reproduce the particular weathering influences that are of interest. It is believed that the characteristics recommended herein cover those points of fundamental importance that will provide an acceptable accelerated weathering unit for many purposes, and at the same time permit the development of detailed and specific cycles needed for the obtaining of particular data and falling within the general structure of this recommended practice. As an example, if the emphasis is on rust-inhibiting paint, in addition to any cycle adopted, it may be desirable to have an accessory high humidity cabinet made available.

The light source shall be a carbon arc. The carbons shall be of such composition and operating under such conditions that the quality of the spectrum as it strikes the test specimens shall approach so far as possible the spectrum of the sun.

The weathering unit shall include means for measuring and controlling the following:

Current, voltage, temperature of air and water, and running time.

The materials of construction shall be of such character as not to react with the test specimens during the test.

Light values shall be measured by the oxalic acid-uranyl sulfate method as follows: A freshly prepared solution of uranoxalate is made by adding 4.2 g. of uranium sulfate to 1 liter of *N*/10 oxalic acid solution. Two quartz tubes, each containing 25 ml. of the uranoxalate solution are supported 6 in. from the arc, one on each side, and exposed to the source of light for 30 min. The solutions are then acidified with sulfuric acid and titrated with *N*/10 KMnO_4 solution at 70° C. The mean of the two readings is taken. The intensity of the arc is measured by the loss in strength of the uranoxalate solution. The original actinic strength of the arc is usually between 40 and 50 per cent, and should not be allowed to fall below 30–35 per cent.

Specimens shall be mounted vertically and shall rotate about the arc in order to provide uniform distribution of the light. If the specimens are mounted vertically both above and below the horizontal center line of the light source, their position should be transposed periodically to provide uniform distribution of the light in

a vertical plane over the entire face of the specimen. Products of combustion shall not be permitted to contact the specimens.

The air temperature at the distance of the surface of the specimens under test shall be constant. It shall be determined by a shielded thermometer at a point most remote from the water spray.

Water sprays shall be provided whereby clean water such as drinking water may be forced on the specimens to simulate the washing action of rain, to provide moisture for causing alternate expansion and contraction due to swelling and drying out, and to introduce thermal shock and sweating. No recirculation or immersion in the spray water shall be permitted.

NOTE.—Certain types of water may contain constituents deleterious to the materials under test.

The accelerated weathering unit at all times shall be operated under constant voltage at the recommended arc amperage. The proper carbons shall be used, the globes and filters cleaned at frequent intervals, and the temperatures of the air and water shall be regulated within the tolerances established.

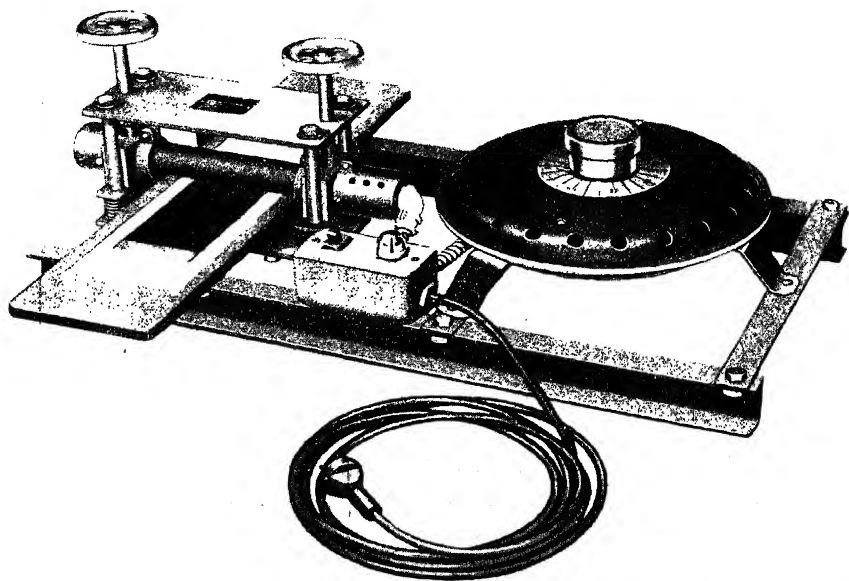
For maintaining a uniform temperature during the light period the weathering apparatus shall be equipped with a thermo-regulator and ventilating equipment which permit automatic maintenance of temperature, or the accelerated weathering machine shall be placed in a room designed to maintain uniform temperature.

Trimmer: The trimmer or "doctor" (Fig. 391) consists of an electrically-heated roll, equipped with thermostatic control, suspended over a stationary base. The roll can be raised or lowered by means of set-screws.

Preparing Test Panels: Specimens shall be prepared as follows:

- (a) Bituminized Fabrics are simply cut to the proper size.⁴⁴
- (b) Bituminous-Solvent Compositions shall be applied to glass or aluminum panels in two or three coats, and should be tested at least in duplicate, to compensate for any irregularities in application.
- (c) Bituminous Substances should be applied to aluminum panels in the following manner: The sample shall be melted at the lowest possible temperature in a seamless can and then poured upon the heated (130 to 160° F., 55 to 70° C.) 3 by 6 by $\frac{1}{8}$ in. aluminum panel. Just prior to pouring, the aluminum panel shall be placed on a piece of folded typewriter paper on the base of the trimmer. The paper takes up excess bituminous material and keeps

the base clean. For convenience it is better to use two such papers so that one remains stationary and the other draws the panel under the trimmer, Fig. 391. The panel and the bituminous material shall then be drawn under the previously set and heated trimmer so that a bituminous film of $0.025 \text{ in.} \pm 0.003 \text{ in.}$ is obtained. It



Courtesy Bureau of Standards

FIG. 391.—The Trimmer for Preparing Panels of Uniform Thickness.

may be necessary to draw the panel back and forth under the heated roll several times. The panel shall then be allowed to cool, the edges shall be cleaned and then calipered, five readings being taken lengthwise. It is important in all these operations that overheating of the bituminous material be avoided. After each coating operation, the trimmer shall be wiped with cloth or paper to keep the roll clean and smooth. The temperature of the trimmer is approximately correct when paper held against it slightly browns.

(1) *Method of Testing Bituminized Fabrics and Bituminous Substances:* The following test procedure has been standardized:⁴⁵

This recommended practice is intended to produce rapid deterioration of bituminous materials under conditions simulating extreme outdoor exposure.

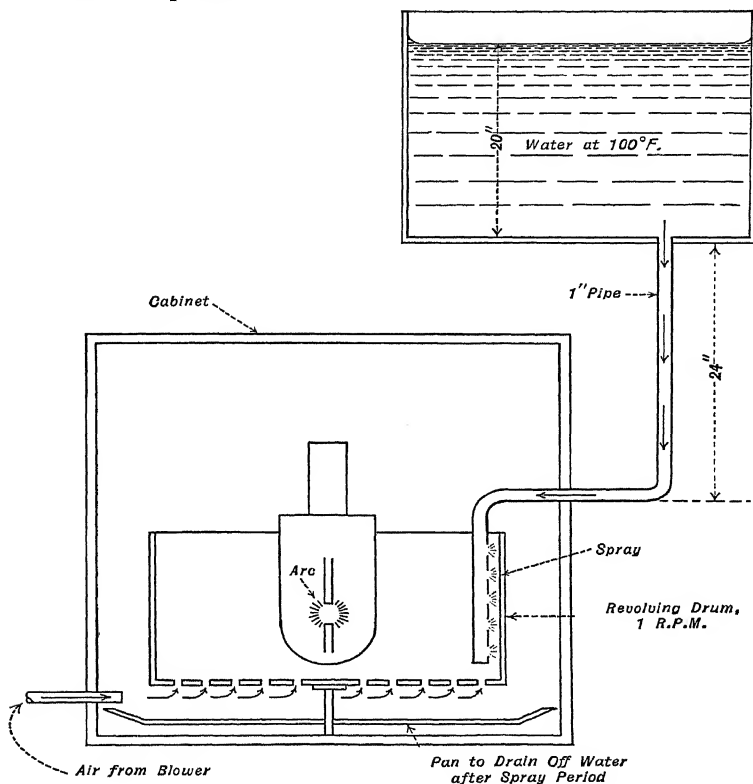


FIG. 392.—Accelerated Weathering Apparatus Provided with Constant Temperature Water Spray.

The apparatus required consists of a constant temperature water reservoir, a cabinet or tank in which are placed an enclosed carbon-arc lamp, a drum, a spray, and an arrangement for temperature control. The general arrangement of the accelerated weathering machine is schematically shown in Fig. 392, which is suitable for testing materials in a vertical position, such as the harder bituminous compounds and roofings. Soft materials, of

necessity, should be tested in a horizontal position, and a mercury-arc lamp is better adapted in that case than a carbon-arc lamp.

(a) Constant Temperature Water Reservoir: The reservoir shall be deep enough to furnish a column of water 20 in. high. A 1-in. pipe, forming an L, shall connect the bottom of the reservoir with the spray. The vertical distance between the spray and the reservoir shall be 2 ft. The water in the reservoir shall be kept at a constant temperature of $100 \pm 5^{\circ}\text{F}$. After each "spray" period, the reservoir is automatically replenished with water and again heated to the desired temperature.

(b) Carbon-arc Lamp: The carbon-arc lamp (Note) shall operate on a 220-v. line, alternating current. The voltage shall be reduced by means of a resistance to 130 to 145 v. The lamp shall operate at 15 to 17 amp., and shall have approximately the following spectral range:

Spectral Range, $\mu\mu$	Percentage Total Radiation to Limit of Fluorite Transmission in Infra Red (12,000 $\mu\mu$) Alternating Current Arc, 60 cycles, 15 to 17 amp.
279 to 290.....	0.8
290 to 320.....	4.1
320 to 360.....	6.0
360 to 480.....	14.5
480 to 600.....	8.0
600 to 1,400.....	14.8
1,400 to 4,200.....	21.4
4,200 to 12,000.....	30.4

Under these conditions the intensity of the light (15 in. opposite the flame) shall be between 3500 to 4000 foot-candles.

NOTE.—The alternating carbon-arc lamp described in this method is taken as the standard as it is now in general use. Mercury-arc, direct-current carbon-arc lamps, and other types of alternating-current carbon-arc lamps if employed must be defined in relation to the standard lamp.

(c) Cylinder: An open, metal cylinder 30 in. in diameter and about 15 in. in depth, equipped with slots or hooks, to hold two tiers of the 3 in. x 6 in. test specimens and connected with a revolving mechanism, geared to furnish 1 rpm. in 20 min., shall surround the lamp. The slots shall be so placed on the inner side of the drum that the point midway between the two rows of panels shall be opposite the center of the arc when the lamp is in opera-

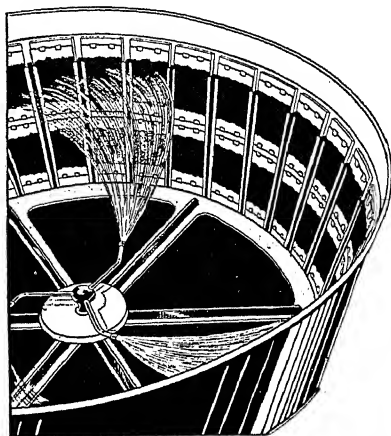
tion. The lamp and the drum shall be suspended over a pan which acts as a drain.

(d) Spray: A spray shall be placed vertically, close to the inner rim of the drum (Fig. 392). The spray is controlled by a solenoid valve. An alternate type of rotary sprinkler is illustrated in Fig. 392A.

(e) Refrigerator: A refrigerator capable of gradually lowering the temperature of the panels from room temperature to 20° F. within 1½ hrs. shall form a separate unit of the accelerated weathering machine.

(f) Temperature Control: The temperature within the cabinet shall be controlled by a bimetallic thermostat located just inside the rows of panels. This thermostat shall be connected to a relay switch which starts a fan blowing air into the cabinet whenever the temperature exceeds that desired. The blowing mechanism shall be so arranged that the fresh air can be directed against the panels from the bottom. The bimetallic thermostat shall be so adjusted that the fan starts blowing air into the cabinet whenever the panels reach a temperature of 140° F. This temperature shall be determined by placing an A.S.T.M. thermometer E 1 (20 F-39), the bulb of which has been coated with a ⅛-in. film of asphalt, ¼ in. in front of the test specimens and at a point where the bulb is opposite the center of the arc. This temperature shall be thermostatically controlled within ±2° F.

The test specimens shall be approximately 6 in. in length and 3 in. in width. Bitumens and bituminous compounds shall be melted and spread to a uniform thickness of 0.025 in. over one surface of an aluminum plate of about ⅛ in. in thickness. Fabricated materials, such as bituminous roofing materials, shall be cut to size and their weather surface exposed directly to the test cycle.



Courtesy A.S.T.M.

FIG. 392A.—Rotary Sprinkler.

The test specimens shall be subjected to a daily weathering cycle consisting of periods of exposure to cold, light, and water.

The weathering cycle (Note) shall be one of the other of the following three types according to the purpose of the test.

NOTE.—Three cycles are offered because experience has taught that weather varies considerably from place to place. The difference between the three cycles is principally in the water treatment; cycle A is a fairly "dry" cycle, cycle B is wetter, while cycle C is the wettest and simulates a hot humid climate. Any of these cycles will determine the effect of the weathering upon bituminous materials.

CYCLE A ^a

	For a period of
Cold exposure.....	1 $\frac{3}{4}$ hr.
Water exposure.....	1 hr.
Light exposure.....	1 $\frac{1}{2}$ hr.
Water exposure.....	2 hr.
Light exposure.....	16 $\frac{1}{2}$ hr.

CYCLE B

Water exposure.....	$\frac{1}{4}$ hr.
Cold exposure.....	1 $\frac{3}{4}$ hr.
Light-spray.....	4 hr.
Water exposure.....	$\frac{1}{2}$ hr.
Light exposure.....	17 hr.

CYCLE C

Light-spray.....	22 hr.
Cold exposure.....	1 $\frac{3}{4}$ hr.

^a To change from one exposure period to the other requires approximately 15 min

For the light, rain, and light-spray exposure periods, the specimens shall be placed in the holders of the cylinder. For the rain period the lamp shall be raised above the cylinder, the latter covered, and the specimens sprinkled vigorously with water by means of a rotary spray which operates at the bottom of the cylinder.

For the cold exposure period, the specimens shall be placed in the refrigerator, previously cooled to -10° F. (-23° C.). To change the panels from the cylinder to the refrigerator shall not require more than 15 min. and the same time may again be allowed after the cold period to return the specimens to the cylinder for the light-spray period.

For the light period, the lamp shall be operated with the revolving cylinder. During the light-spray period, the light from the lamp shall shine on the specimens continuously while the water,

running constantly from the spray at a fixed point, shall wet the specimens in the revolving cylinder once every 20 min.

The air temperature during the light period shall be $140 \pm 5^{\circ}\text{F.}$ ($60 \pm 2.8^{\circ}\text{C.}$). This temperature shall be measured by means of a transparent-bulb, mercury thermometer placed behind the aluminum panel holder so that the bulb of the thermometer is shielded from the arc light by the panel holder between the two tiers of test specimens and shall be half way between the panel holder and the cylinder of the weathering apparatus.

The radiation temperature during the light period shall be $180 \pm 5^{\circ}\text{F.}$ ($82 \pm 2.8^{\circ}\text{C.}$). This temperature shall be measured by means of a mercury thermometer, the bulb of which is thinly coated with unweathered asphalt. This thermometer shall be mounted inside the rotating cylinder at a distance of $\frac{1}{4}$ in. from the surface of the center of the panels and with its bulb on a level with the center of the panel holder between the two tiers of test specimens.

In the case of the light-spray period, the specimens will, of course, be cooled as they pass by the water spray; the specimens should, however, warm to about these standard temperatures before being wet again. (At times it may be necessary to use warm water for spraying in order to reach such a temperature.) Water (2 to 4 in. in depth) shall be kept in the tank or sump all the time, the excess water flowing out over the overflow.

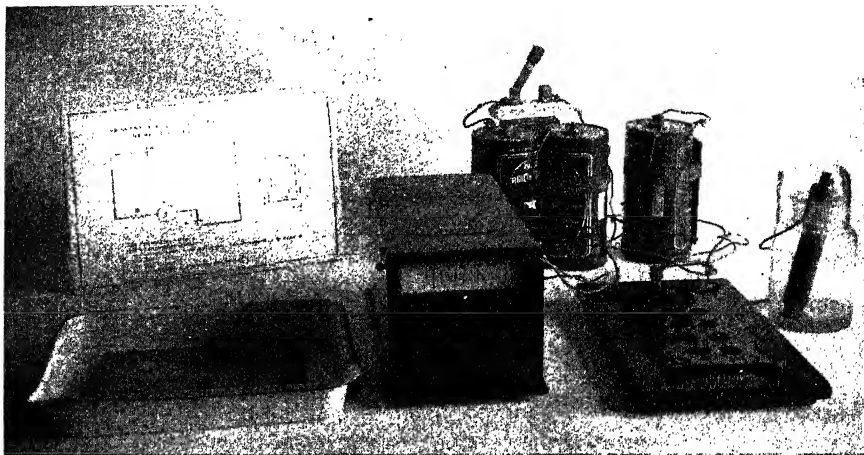
The position of the panels in the revolving cylinder shall be changed for each repetition of the cycle, with respect to their location in the upper or lower tiers of slots or hooks, in order to compensate for unavoidable differences in their relation to the light source.

Determining Extent of Weathering: The result of the accelerated weathering test shall be determined by comparing specimens of the weathered material with test specimens that have not been weathered. The durability of the material may be expressed as the number of cycles of weathering that are required to bring about definite changes in the pliability, breaking strength, solubility in standard solvents, and appearance of the material tested.

The principal test is the estimation of the surface differences

before and after weathering. This may be carried out on instruments of the photo-electric type. All these tests are general but for bituminous materials surfaced on aluminum panels, however, the "conductivity test" (Fig. 393) for determining the extent of weathering shall be made as follows:

The apparatus consists of two dry-cells, a 0.5 megohm resistance, a galvanometer, an electrolyte cell, and the asphalt panel,



Courtesy Bureau of Standards

FIG. 393.—Conductivity Test Apparatus.

connected in series. The electrolyte cell contains a 2 per cent solution of magnesium chloride in a 5 per cent alcohol solution. The electrolyte is applied to the asphalt surface by means of a short, round camel's hair brush. If any cracks extend through the asphalt to the aluminum panel, a galvanometer-needle deflection is obtained. Panels are considered to have failed when two deflections are obtained more than $\frac{1}{4}$ in. from the edges.

The weathering test shall be considered completed when six or more positive tests (out of a possible ten) are obtained. The test shall be reported as the number of weathering cycles necessary to obtain this result. For permanent records the test specimens shall be photographed. A Leica or a fingerprint camera is suitable for this purpose.

(II) *Method of Testing Bituminous Preformed Expansion Joints:* The following procedure has been standardized for examining preformed expansion-joints:⁴⁶

Two specimens, prepared as described, shall be exposed to a temperature of 165° F. (74° C.) for a period of 7 days. Upon completion of this accelerated aging test, the specimens shall be immersed in water at room temperature for 24 hrs.

The specimens shall then be placed on edge in a suitable container and water poured into the container to a depth of 2 in. (one-half the height of the specimens). It will be necessary to put a weight or simple frame across the exposed edges of the specimens during this test in order that the positions of the specimens in the water will be maintained. The pan containing the specimens partially immersed in water shall be placed in a freezing chamber for a period long enough to freeze the water into solid ice. The temperature of the freezing chamber shall be maintained between +14 and -4° F. (-10 and -20° C.). Upon completion of the freezing cycle, the pan containing the specimens shall be removed from the freezing chamber and partially immersed in water at a temperature maintained between 65 and 100° F. (18 and 38° C.). The first cycle is completed when the ice surrounding the specimens has melted entirely. This cycle shall be repeated ten times.

The length of time required for freezing and thawing will be governed by the temperature of the freezing chamber and the volume of water around the specimens.

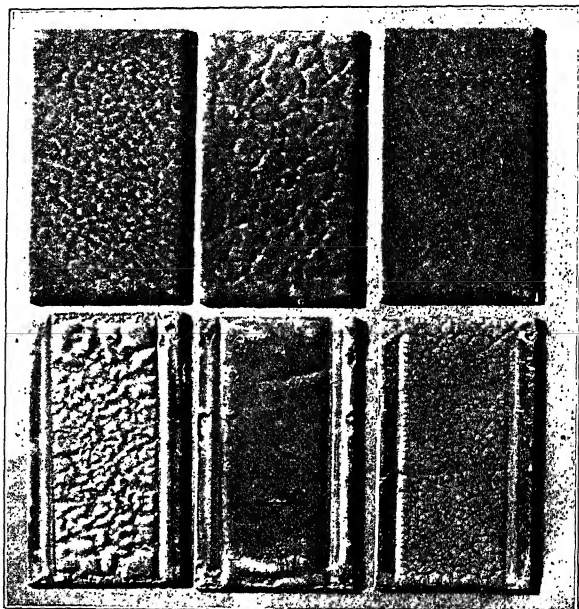
After ten freezing-and-thawing cycles have been completed, the specimens shall be removed from the water and allowed to stand in air at room temperature for 48 hrs. The test specimens shall be examined for evidences of disintegration.

(III) *Method of Testing Bituminous-solvent Compositions:* It has been proposed that in the case of bituminous-solvent compositions, the exposed specimen be bent double in a predetermined time at a prescribed temperature over a rod of a fixed diameter. It is contended that the more durable the composition, the longer it may be exposed to the ultra-violet light before it will crack on bending.⁴⁷

Figure 394 illustrates the appearance of typical asphalts, all having a fusing-point of 220° F. (R. and B. method), the upper

row having been exposed out-of-doors for one year under quartz glass (Test 101), and the lower row, representing the identical asphalts, after having been subjected to the accelerated weathering cycle (Test 102) for 70 cycles.

Table CLIX shows the behavior of blown asphalts, with and without the addition of mineral fillers to the accelerated exposure



Courtesy Bureau of Standards

FIG. 394.—Weathering of Typical Asphalts. Upper row: exposed one year out-of-doors. Lower row: shows the same asphalts after being subjected to the weathering cycle for 70 days.

test, the final appearances being illustrated in Fig. 395. Of the unfilled asphalts tested, Mid-continental stood first, Californian and Colombian second, and Texas third, in the order of their respective weather-resistance. Of the fillers tested, alberene flour (fine-mesh) stood first, slate flour stood second, talc third, limestone flour fourth, silica dust and dolomite flour fifth, silica sand and hydrated lime sixth. With any particular filler, the finer the mesh, the better proved to be the resistance.

TABLE CLIX

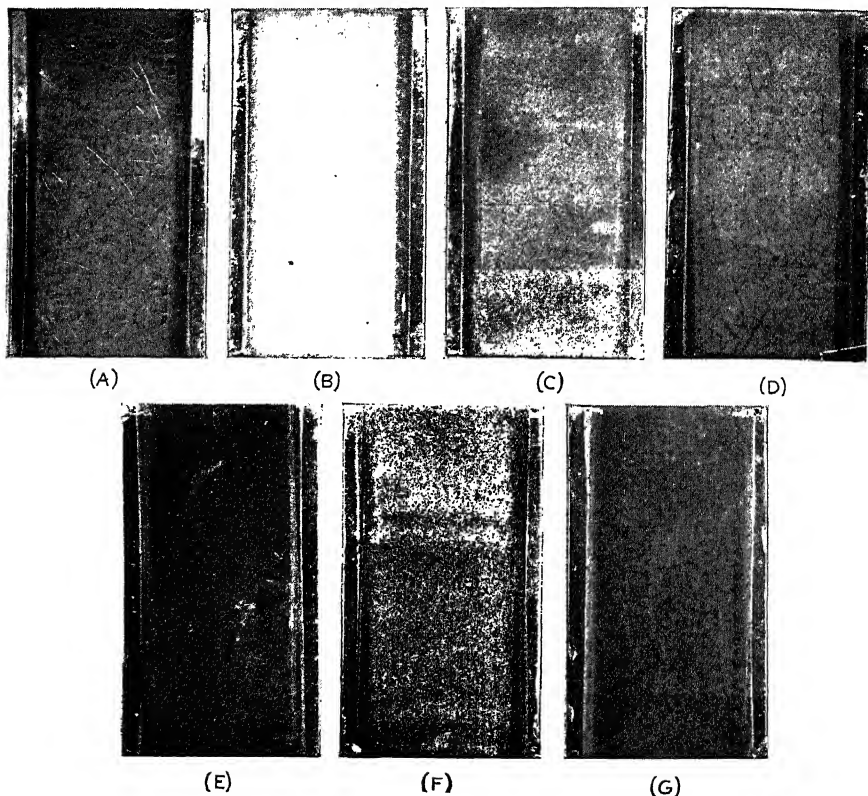
ACCELERATED WEATHERING TESTS—BLOWN PETROLEUM ASPHALTS

Figure 395	Blown Asphalt R. and B. Fusing-pt. °F.	Mineral Filler Added (35% by Weight)	No Cracks Visible to the Eye	Cracks Visible; Aluminium Panel Not Exposed	Cracked through; Aluminium Panel Visible (= Failure)
<i>Produced from California Petroleum:</i>					
(A)	221	None.....	<i>Cycles</i> Below 40	<i>Cycles</i>	<i>Cycles</i> At 41-42
(B)	221	Alberene flour—350 mesh.	Below 101 (*)
...	221	“ “ —200-300 mesh.	Below 88	At 93(†)
...	221	“ “ —150-200 mesh.	Below 88	From 93 to 101(*)
...	221	“ “ —100-150 mesh.	Below 58	At 61	At 76
(C)	221	Fine silica sand.....	Below 41	At 42
(D)	221	Silica dust.....	Below 56	At 58
(E)	221	Hydrated lime.....	Below 41	At 42
(F)	221	Talc (Georgia).....	Below 76	From 81 to 101(*)
<i>Produced from Mid-Continental Petroleum:</i>					
(G)	225	None.....	Below 101 (*)		
<i>Produced from Texas Petroleum:</i>					
(H)	219	None.....	Below 30	At 37	At 42
(I)	225.5	Alberene flour (200-300 mesh.)	Below 30	From 37 to 50(*)
(J)	223.7	Dolomite flour.....	Below 30	At 37	At 46
<i>Produced from Colombian Petroleum:</i>					
...	225	None.....	Below 39	At 40-42
(K)	225	Slate flour.....	Below 84(*)
...	225	Limestone flour.....	Below 64	From 71 to 84(*)
(L)	225	Dolomite flour.....	Below 54	From 59 to 84(*)
(M)	225	Hydrated lime.....	Below 30	At 39	At 43

(*) Test discontinued.

(†) Observation not conclusive.

Investigations have also been made ⁴⁸ to show the effect of mineral fillers on the durability of asphalt coatings by exposing samples outdoors and to the accelerated weathering cycle. The same type

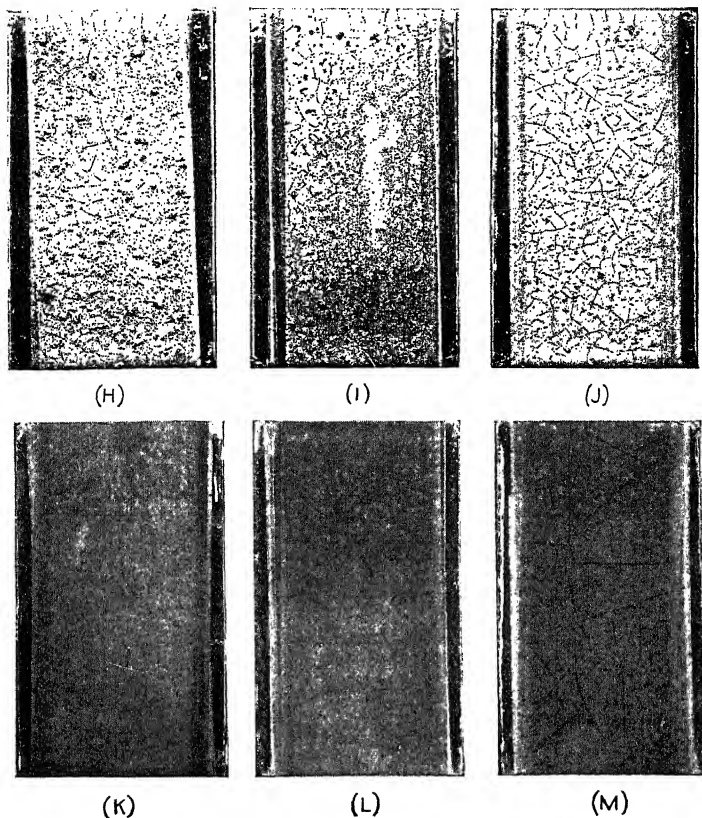


Courtesy Bureau of Standards

FIG. 395.—Accelerated Weathering of Blown Petroleum Asphalts.

of asphalt, made from the same crude in a single operation, but differing only in the duration of the blowing, was prepared with softening-points (R. and B. method) of 194° and 225° F. respectively. These two asphalts were so blended that, when combined with the stipulated weight of filler, the softening-point of the re-

sulting mixture was 225° F. The compositions and physical properties of the various compositions tested are shown in Table CLX. The physical properties of the fillers themselves are given in Table



Courtesy Bureau of Standards

FIG. 395.—Accelerated Weathering of Blown Petroleum Asphalts (Continued).

CLXI, in which the “compacting weight” represents the weight of the filler per milliliter when thoroughly compacted by tapping, and the “fineness factor” (“*f*”) is calculated by subtracting the compacting weight from the density and dividing the result by the compacting weight.

TABLE CLX

COMPOSITIONS, PHYSICAL PROPERTIES, AND DURABILITIES OF ASPHALT-FILLER MIXTURES

Specimen	Asphalt-Filler Mixture, per cent			Percentage of Hard Asphalt in Total Asphalt	Kind of Filler	Penetrations in 0.01 cm. units ^a			Durability	
	Hard Asphalt	Soft Asphalt	Filler			At 32 F., 200 g., 60 sec.	At 77 F., 100 g., 5 sec.	At 115 F., 50 g., 5 sec.	1½ yr. Outdoor Exposure, Group Classification ^b	Life in Accelerated Test cycles
No. 1...	47	18	35	72	Green slate flour, filler No. 1	6	9	13	No. I	62
No. 2...	62	13	25	86		6	9	16	No. II	45
No. 3...	73	12	15	86		7	9	17	No. III	37
No. 4...	47	18	35	72	Dolomite, filler No. 2	6	10	18	No. II	37
No. 5...	50	15	35	80		6	10	17	No. III	28
No. 6...	73	12	15	86		7	10	17	No. III	28
No. 7...	47	18	35	72	Dolomite, filler No. 3	6	9	15	No. II	40
No. 8...	60	15	25	80		8	11	18	No. III	28
No. 9...	73	12	15	86		7	10	17	No. III	28
No. 10...	47	18	35	72	Limestone, filler No. 4	6	9	16	No. II	37
No. 11...	60	15	25	80		6	10	17	No. III	43
No. 12...	73	12	15	86		6	9	18	No. III	33
No. 13...	47	18	35	72	Peach-bottom slate, filler No. 5	6	8	15	No. I	65
No. 14...	60	15	25	80		7	10	16	No. II	62
No. 15...	73	12	15	86		8	11	18	No. III	43
No. 16...	47	18	35	72	Silica sand, filler No. 6	8	12	18	No. II	28
No. 17...	60	7	25	90		11	11	15	No. II	28
No. 18...	80	3	15	94		11	11	14	No. III	28
No. 19...	47	18	35	72	Greenstone, filler No. 7	7	11	14	No. I	65
No. 20...	60	15	25	80		7	10	16	No. II	40
No. 21...	73	12	15	86		7	10	17	No. III	28
No. 22...	—	65	35	0	Hydrated lime, filler No. 8	6	8	14	No. III	28
No. 23...	—	75	25	0		8	11	21	No. III	28
No. 24...	37	48	15	56		7	11	21	No. III	28
No. 25...	36	29	35	55	Trap rock, filler No. 9	6	9	15	No. II	37
No. 26...	53	22	25	70		7	9	17	No. III	43
No. 27...	72	13	15	85		7	9	16	No. III	28
No. 28...	13	72	15	15	Supercel, filler No. 10	8	10	22	No. I	40
No. 29...	41	44	15	48	Mica, filler No. 11	7	10	17	No. I	65+
No. 30...	23	42	35	36	Foliated talc, filler No. 12	6	10	16	No. I	65
No. 31...	44	31	25	58		7	9	18	No. II	65
No. 32...	66	19	15	78		7	9	18	No. II	48
No. 33...	49	16	35	75	Silica dust, filler No. 13	7	8	16	No. II	35
No. 34...	62	13	25	83		7	8	16	No. II	33
No. 35...	78	7	15	92		7	9	17	No. III	28
No. 36...	40	25	35	62	Silica dust, filler No. 14	7	9	18	No. II	35
No. 37...	54	21	25	72		6	9	16	No. III	28
No. 38...	72	13	15	85		6	9	16	No. III	28
No. 39...	49	16	35	75	Silica dust, filler No. 15	7	9	17	No. II	35
No. 40...	64	11	25	85		7	10	17	No. II	35
No. 41...	77	8	15	91		7	9	17	No. III	28
No. 42...	33	32	35	51	Slate flour, filler No. 16	7	10	17	No. I	65
No. 43...	50	25	25	67		7	10	17	No. II	51
No. 44...	68	17	15	80		7	10	17	No. II	43
No. 45...	44	21	35	68	Slate flour, filler No. 17	7	10	14	No. I	65
No. 46...	58	17	25	78		7	10	15	No. I	65
No. 47...	75	10	15	88		7	10	18	No. II	43
No. 48...	43	22	35	66	Limestone, filler No. 18	7	10	18	No. II	45
No. 49...	50	19	25	75		7	10	18	No. III	33
No. 50...	75	10	15	88		7	10	19	No. III	28
No. 51...	49	16	35	75	Limestone, filler No. 19	7	10	18	No. II	58
No. 52...	64	11	25	85		8	10	17	No. II	48
No. 53...	81	4	15	95		6	10	19	No. III	28
No. 54...	100	—	—	100	7	10	17	No. III	28
No. 55...	75	25	—	75	9	11	17	No. III	28
No. 56...	50	50	—	50	8	12	21	No. III	28
No. 57...	25	75	—	25	9	15	27	No. III	28

^a The results reported are the average of 3 or 4 determinations.^b Group No. I.—Outdoor panels showing no cracks to the eye or with magnifying glass, or, at most, some of the panels of a set showing faint cracks to the eye or with a magnifying glass. Group No. II.—Outdoor panels showing cracks to the eye. Group No. III.—Outdoor panels showing wide cracks to the eye.

TABLE CLXI
PHYSICAL PROPERTIES OF THE FILLERS

Filler	Kind of Filler	Particle Size of Filler			Oil-Absorption Ratio	Specific Gravity	Compacting Weight	Fitness Factor <i>f</i>
		Passing No. 200 Sieve, per cent	Passing No. 100 but Retained on No. 200 Sieve, per cent	Retained on No. 100 Sieve, per cent				
No. 1...	Slate flour—Air floated No. 000 grade.....	81	17	1	0.7	2.86	1.35	1.1
No. 2...	Dolomite.....	92	7	1	0.5	2.85	1.63	0.8
No. 3...	Dolomite.....	85	14	1	0.4	2.88	1.73	0.7
No. 4...	Limestone.....	91	8	1	0.4	2.76	1.62	0.7
No. 5...	Peach-bottom slate—No 000 grade.....	79	16	5	0.8	2.98	1.40	1.1
No. 6...	Silica sand.....	2	13	85	0.2a	2.69	1.74	0.6
No. 7...	Greenstone.....	65	24	12	0.6	3.08	1.86	0.9
No. 8...	Hydrated lime.....	99	2	1.3	2.39	0.83	1.9
No. 9...	Trap rock.....	97	3	0.7	2.94	1.32	0.9
No. 10...	Celite (Hy Flo Supercel).....	100	2.9	2.52	0.30	7.4
No. 11...	Mica.....	42	28	30	2.2b	3.20	0.51	5.3
No. 12...	Foliated talc.....	100	trace retained on No. 200 sieve	1.1b	2.97	1.05	1.8
No. 13...	Silica dust.....	67	26	7	0.5b	2.69	1.68	0.6
No. 14...	Silica dust (prepared from filler No. 13).....	100	0.5	2.69	1.63	0.7
No. 15...	Silica dust (prepared from filler No. 13).....	100	0.4	2.69	c
No. 16...	Slate flour (prepared from filler No. 5).....	100	0.9	2.98	1.30	1.3
No. 17...	Slate flour (prepared from filler No. 5).....	100	0.7	2.98	c
No. 18...	Limestone (prepared from filler No. 4).....	100	0.5	2.76	1.63	0.7
No. 19...	Limestone (prepared from filler No. 4).....	100	0.5	2.76	c

^a Does not possess plastic properties, but merely wets.

^b Not a sharp end-point.

^c Insufficient material for test.

The outdoor panels were exposed to the weather for 1½ years, whereupon they were examined for cracks visually and under a magnification of ×12. Duplicate panels were subjected to the standard accelerated weathering test and the durability of the respective coatings expressed in cycles. It will be observed that in general the coatings failed in the same order in the outdoor tests as they did in the accelerated cycle, and it can be reasonably assumed that 30 cycles in the accelerated test are equivalent to about 1½ years' outdoor exposure. The life of the coatings in the accelerated test is given in cycles in Table CLXII.

Test 103. Modified Accelerated Weathering Test, Based on Increase of Water-soluble Constituents. The Bureau of Standards, Washington, D. C., has recommended a modification which

TABLE CLXII
ORDER OF DURABILITY OF ASPHALT-FILLER MIXTURES

Specimen Number	Composition			Durability (Cycles)
	Amount (Per Cent)	Kind of Filler	Filler No.	
GROUP I				
29	15	Mica.....	11	65+
30	35	Talc.....	12	65
31	25	Talc.....	12	65
42	35	Slate.....	16	65
45	35	Slate.....	17	65
13	35	Slate.....	5	65
1	35	Slate.....	1	62
14	25	Slate.....	5	62
46	25	Slate.....	17	65
43	25	Slate.....	16	51
32	15	Talc.....	12	48
GROUP II				
51	35	Limestone.....	19	58
19	35	Greenstone.....	7	65
39	35	Silica.....	15	35
40	25	Silica.....	15	35
2	25	Slate.....	1	45
47	15	Slate.....	17	43
28	15	Supercel.....	19	40
20	25	Greenstone.....	7	46
36	35	Silica.....	14	35
44	15	Slate.....	16	43
25	35	Trap rock.....	9	43
16	35	Silica.....	6	28
17	25	Silica.....	6	28
41	15	Silica.....	15	28
52	25	Limestone.....	19	48
5	25	Dolomite.....	2	28
15	15	Slate.....	5	43
4	35	Dolomite.....	2	37
3	15	Slate.....	1	37
33	35	Silica.....	13	35
34	25	Silica.....	13	33
26	25	Trap rock.....	9	37
8	25	Dolomite.....	3	37
37	25	Silica.....	14	30
7	35	Dolomite.....	3	40
11	25	Limestone.....	4	37
48	35	Limestone.....	18	45
10	35	Limestone.....	4	43
21	15	Greenstone.....	7	40
GROUP III				
18	15	Silica.....	6	28
38	15	Silica.....	14	28
27	15	Trap rock.....	9	28
35	15	Silica.....	13	28
9	15	Dolomite.....	3	28
53	15	Limestone.....	19	28
54	Asphalt, softening point 108° C.....	28
55	Asphalt, softening point 104° C.....	28
56	Asphalt, softening point 99.5° C.....	28
57	Asphalt, softening point 94° C.....	28
49	25	Limestone.....	18	33
50	15	Limestone.....	18	28
6	15	Dolomite.....	2	28
12	15	Limestone.....	4	33
24	15	Hydrated lime.....	8	28
23	25	Hydrated lime.....	8	28
22	35	Hydrated lime.....	8	28

consists in exposing a series of panels aggregating about 180 sq. in. of surface to the carbon arc for 18 hrs., followed by immersion of the panels in distilled water at room temperature for 5 to 6 hrs. After periods of 26 cycles, the water solution containing the soluble decomposition products is evaporated to dryness at 105° C. and the residue weighed. The results recorded in Table CLXIII-A have been reported:⁴⁹

TABLE CLXIII-A

BEHAVIOR OF BLOWN PETROLEUM-ASPHALTS IN STANDARD AND MODIFIED ACCELERATED TESTS

Blown Asphalt (R. and B. fusing-point 108° C.)	From Mid- Continental Petroleum	From Mexican Petroleum	From Venezuelan Petroleum	From Californian Petroleum
<i>Standard Accelerated Test:</i>				
Life in cycles.....	56	26	34	29
Appearance at end of test.....	Smooth	Pronounced hummocks	Hummocks	Pronounced hummocks
<i>Modified Accelerated Test (a):</i>				
Life in cycles.....	179 (b)	87	47	57
Appearance at end of test.....	Smooth; some grooving	Smooth	Smooth; slight grooving	Smooth; some grooving
Water-soluble Products (grams per 100 sq. in. surface):				
1st—26 cycles (Total 26 cycles)	1.161	1.317	1.350	1.583
2nd—26 cycles (Total 52 cycles)	1.311	1.311	1.606	1.694
.... 9 cycles (Total 61 cycles)	0.500	0.444	0.400	0.655
3rd—26 cycles (Total 87 cycles)	1.233	1.322	1.667	2.411
4th—26 cycles (Total 113 cycles).....	0.778	0.889	1.167	1.722
5th—26 cycles (Total 139 cycles).....	0.667	0.722	1.000	1.222
6th—26 cycles (Total 165 cycles).....	0.556	0.556	0.778	1.167
7th—26 cycles (Total 191 cycles).....	0.500 (c)	(d)	(d)	(d)
Total in 165 cycles.....	6.206	6.561	7.968	10.454
Appearance at end of 191 cycles..	Asphalt layer intact	Slight loss of asphalt layer	Slight loss of asphalt layer	About third of asphalt layer lost

(a) 12 panels of each specimen subjected to Modified Accelerated Test, exposing about 180 sq. in. surface of asphalt, weight about 140 grams.

(b) Started to crack at 125 cycles.

(c) Not included in total of 6.206.

(d) Lost adhesiveness and fell off panel.

Fig. 396 shows the appearance of the panels at the end of the standard accelerated test, Fig. 397 shows the appearance of the specimens at the end of 165 modified accelerated cycles, and Fig. 398 at the end of 191 modified accelerated cycles.

Further investigations on these same specimens, including the soluble portion of Trinidad asphalt, revealed the results given in Table CLXIII-B.

Test 104. Modified Accelerated Weathering Test, Based on Increase in Pentane-insoluble Constituents.

The progress of the accelerated test may be followed by means of Test 38*c*, Test 38*d* and Test 38*e*. Thus, a blown petroleum asphalt having a fusing-point of 220° F. behaved as follows:

Cycles	Asphaltenes, %	Asphaltic Resins, %	Oily Constituents, %
Originally.....	26.2	4.8	68.0
After 21 cycles.....	34.6	1.2	61.4
After 80 cycles.....	40.6	1.2	56.6

It will be noted that the asphaltenes increase progressively at the expense of the oily constituents and to a lesser extent at the expense of the asphaltic resins. This is corroborated by heating at 200° C. in an atmosphere of oxygen for 3 hours. Asphaltenes became 96 per cent insoluble in carbon disulfide and asphaltic resins produced a product which was 31 per cent insoluble.

A further investigation consisted in first separating petroleum asphalts into asphaltenes, asphaltic resins and oily constituents, which components were then recombined in various proportions. The synthetic asphalts, thus produced, were subjected to accelerated weathering tests, and the observations indicated: (a) that increased weather-resistance was obtained with increased proportions of asphaltic resins, and (b) that a combination of the oily constituents derived from steam-reduced asphalts when combined with the asphaltenes derived from air-blown asphalts weather better than a mixture of such constituents derived from a straight steam-reduced asphalt.⁵⁰

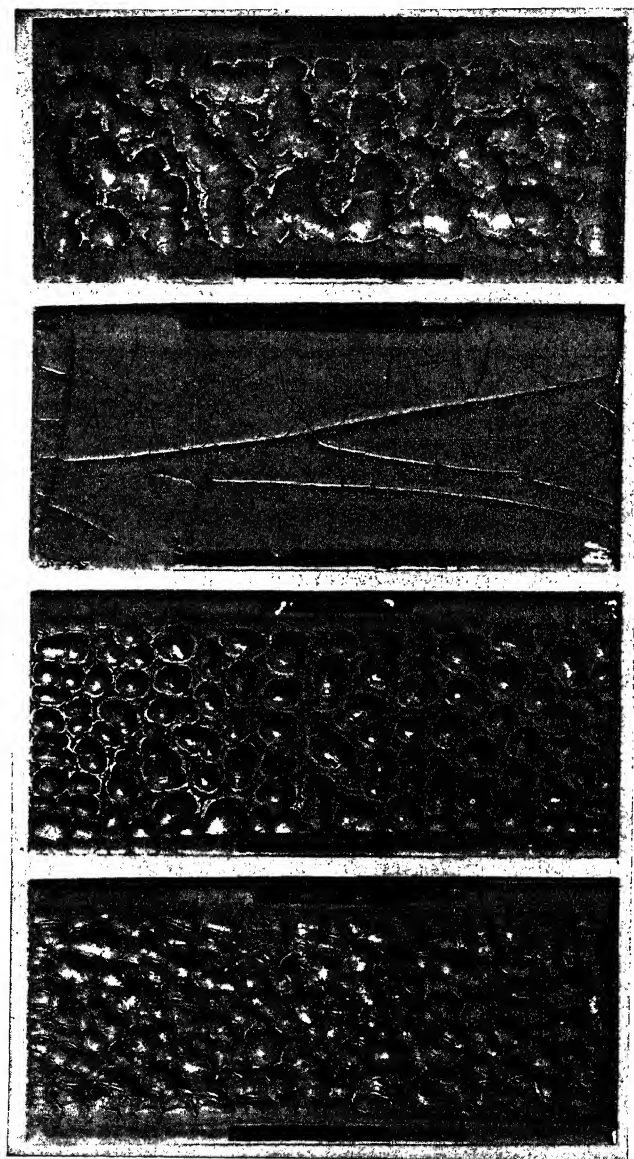
The photo-oxidation of asphalt has been investigated by exposing specimens of the asphalt, as well as its components (i.e., asphaltenes separated by means of pentane; waxes by means of methyl-ethyl-ketone and benzol solution; paraffinic oils and naphthenic oils

TABLE CLXIII-B

BEHAVIOR OF BLOWN PETROLEUM ASPHALTS IN MODIFIED ACCELERATED TEST

Blown Asphalt (R. and B. fusing-point about 108° C.)	From Mid- Contl. Petro- leum	From Mexi- can Petro- leum	From Vene- zuelan Petro- leum	From Califor- nian Petro- leum	From Trinidad Asphalt (soluble portion)
<i>Original Specimen:</i>					
Asphaltenes (insoluble in pentane)....%	36.7	48.3	40.1	44.9
Petrolenes { Oily constituents....%	40.0	33.2	41.0	39.0
(Sol in pentane) { Asphaltic resins....%	22.7	17.8	18.2	15.5
Total.....%	99.4	99.3	99.3	99.4
Loss.....%	0.6	0.7	0.7	0.6
<i>Asphaltenes (insoluble in pentane):</i>					
Formation of products insol. in CCl ₄ after: 173 hours' exposure to arc-light....%	3.0	8.7	7.3	8.8
<i>Petrolenes (soluble in pentane):</i>					
Formation of asphaltenes after: 173 hours' exposure to arc-light....%	0.0	20.7	17.1	20.4	15.9
Formation of products insol. in benzol after: 173 hours' exposure to arc-light....%	0.18	0.38	0.38	0.62
Formation of products insol. in CCl ₄ after: 173 hours' exposure to arc-light....%	0.19	0.71	0.76	1.11	1.50
Amount of same insoluble in water...%	90.9	55.8	71.7	70.1	63.6
<i>Oily Constituents:</i>					
Formation of asphaltenes (9.6 sq. in. sur- face) after: 134 cycles of standard test.....%	0.0	9.1	5.1	11.3
173 hours' exposure to arc-light....%	0.0	13.2	12.9	12.2	15.1
Ml. N/10 KOH required to neutralize the water-soluble constituents after: 320 hours' exposure to arc-light....Ml.	0.3	1.0	0.9	1.5
Formation of products insol. in benzol after: 173 hours' exposure to arc-light....%	0.13	0.58	0.41	0.50
Formation of products insol. in CCl ₄ after: 173 hours' exposure to arc-light....%	0.35	0.55	0.47	0.64	1.4
Amount of same insoluble in water...%	88.7	56.2	73.3	71.8	52.2

by extraction with acetone) in oxygen-filled, sealed flasks under an ultraviolet lamp for a period of 55 hrs. at 165 to 180° F. The net volume of oxygen absorbed per gram of asphalt was measured, and furnished an index of the sensitivity to photo-oxidation. It was found that Mexican residual asphalt absorbed most oxygen, U. S.



Courtesy Bureau of Standards

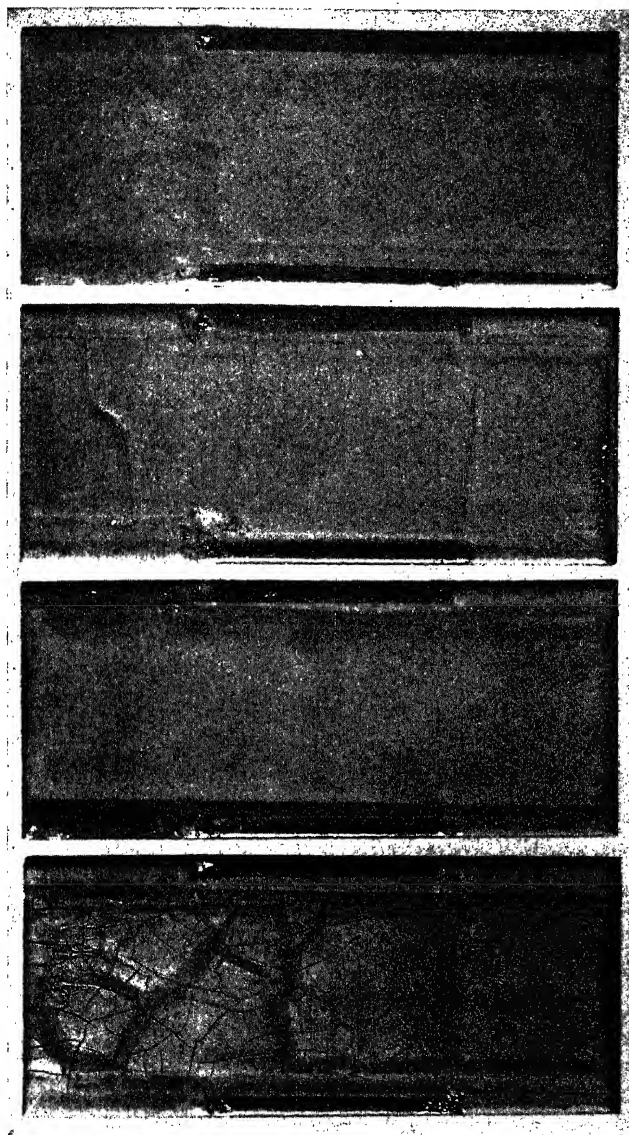
California

Mid-Continental

Mexican

Venezuelan

FIG. 396.—Standard Accelerated Weathering Cycles. Appearance at end of test.



Courtesy Bureau of Standards

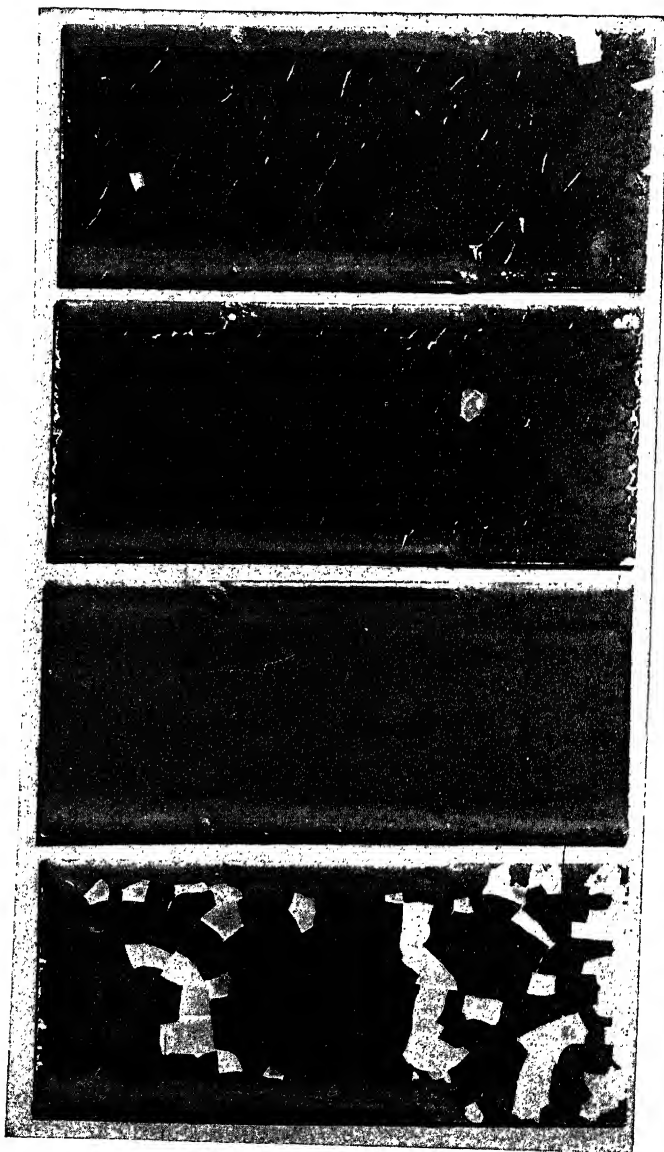
Californian

Mid-Continental

Mexican

Venezuelan

FIG. 397.—Exposed for 165 Modified Cycles.



Venezuelan

Mexican

Mid-Continental

Courtesy Bureau of Standards
Californian

FIG. 398.—Exposed for 191 Modified Cycles.

mid-continental asphalts came next, and Gulf-coast residual asphalt absorbed least, of the three groups tested.⁵¹

An alternate procedure consists in subjecting a solution of the asphalt (100 g.) in benzol (67 g.) to the action of oxygen at 100 lbs. per sq. in. at 50° C., and measuring the drop in pressure over a period of 40 hours. The pressure drop is then plotted against the time. From this graph, the pressure-drop at 40 hrs. and the slope of the tangent at this point (lbs. per sq. in. per hr.) are evaluated, and the product of the pressure-drop and the slope (i.e., rate of oxygen consumption) is recorded as a measure of the "oxidation rating." The asphalt is then recovered from the benzol solution and its penetration recorded at 77° F. The log of this penetration is plotted against the pressure-drop, and the amount of oxygen required to reduce the penetration to 20 is designated the "hardening rating" (expressed in lbs. per sq. in.). From the foregoing data the "deterioration index" is calculated from the following formula:⁵²

$$\text{Deterioration index} = \frac{\text{Oxidation rating}}{\text{Hardening rating}} \times 100$$

Further investigations revealed the following:⁵³ the "oily constituents" separated by Method I of Test 38f were exposed in a weatherometer (5 g. placed in a glass petri dish) for 15 cycles (345 hrs.), thereupon treated with pentane and filtered through a Gooch crucible to separate the pentane-insoluble constituents formed during exposure to the light. The residue in the crucible was washed with water at 70° C., using a vacuum, to ascertain the amount of water-soluble products formed in the oils. The "resins with difference resins" separated by Method I were similarly subjected to light, thereupon first extracted with ethyl-ether to separate the ether-insoluble constituents, followed by water at 70° C. The following results were obtained from 34 representative specimens of blown petroleum asphalt produced in the United States (used for coating asphalt roofings and shingles), after exposure to 15 cycles of light:

Oily constituents (Test 38f—Method I):

Pentane-insoluble.....	0.59 to 22.37 per cent
Water-soluble.....	0.02 to 0.58 per cent

Resins with difference resins (Test 38f—Method I):

Ether-insoluble	0.57 to 1.00 per cent
Water-soluble	0.06 to 0.24 per cent

This indicates that the oily constituents present in the asphalt are largely responsible for its weathering characteristics, since they vary considerably in their resistance to light, whereas the resinous constituents are comparatively little affected.

Further tests made on the same 34 asphalts gave the following results:

Accelerated Test:	Best Specimens	Poorest Specimens
Incr. pentane-insol. 190 cycles..	25.0 to 33.8 per cent	34.2 to 46.8 per cent
Total life in weatherometer....	90 to 190 cycles	60 to 90 cycles
Brittleness on cutting panels with a pair of shears.....	Not brittle to slightly brittle	Brittle (chips)
Outdoor Exposure:		
Incr. pentane-insol. 1 yr. 7 mos..	9.3 to 13.1 per cent	13.9 to 18.4 per cent
Total life in weatherometer....	60 to 190 cycles	65 to 85 cycles

The following simplified procedure has been proposed: Ten or more panels of each specimen are prepared by either of the following alternatives:

Method I: Place an amalgamated brass mask (1.25 by 2.75 in., and 0.025 ± 0.001 in. thick, with rims 0.5 in. wide) over a tared aluminum panel (1.5 by 3.0 in. and 0.03 ± 0.001 in. thick). Melt the asphalt and pour it on the aluminum panel, allow it to cool and then with a heated spatula shave off the asphalt flush with the mask, obtaining a uniform film 0.025 ± 0.003 in. thick.

Method II: Melt the asphalt in a lid of a small seamless tin can and pour to a uniform film 0.025 ± 0.003 in. thick on a tared, slightly preheated aluminum panel (without using the mask); the amount of asphalt being required is calculated from the following formula:

$$\text{Weight asphalt} = 67.7 \times 0.025 \times \text{Sp. gr. of asphalt}$$

The melted asphalt is uniformly spread over the surface of the panel by means of a thin wire, then allowed to cool and reweighed.

With either method, the weight of the asphalt is as follows:

$$\text{Weight coated panel} - \text{Weight bare panel} = \text{Weight asphalt} \quad (I)$$

Subject the panels to the accelerated test consisting of 23 hrs. of light and water-spray a day, representing one cycle, until failure. The water-spray is allowed to run continuously at a fixed point, thereby wetting the specimen in the revolving cylinder once every 20 min. After every 15 cycles of exposure, remove 2 of the panels (for duplicate tests), dry in an oven for 18 hrs. at 70° C., cool in a dessicator and weigh.

Weight of weathered coated-panel (II)

Find the pentane-insoluble matter by placing the panel in a 400-ml. wide-mouthed Erlenmeyer flask, cover with n-pentane and allow to soak for 2 hrs., using a wire or spatula if necessary. Remove the aluminum panel from the flask, immerse in benzol to remove any adhering asphalt and reweigh.

Weight of decoated aluminum panel (III)

Add more pentane to the flask until the volume is 250-300 ml., cork the flask and permit it to settle overnight. Filter out the pentane-insoluble matter in a tared Gooch crucible, using light suction, dry in an oven at 105° C., cool in a desiccator and weigh.

Weight of Gooch containing insol. matter — Weight of Gooch
= Weight of pentane insol. matter collected on Gooch cru-
cible (IV)

Dissolve the residue adhering to the Erlenmeyer flask in a small volume of benzol and transfer this, together with the washings of the aluminum panel described above, to a tared 100-ml. beaker, evaporate, dry in an oven at 105° C., cool in a desiccator and weigh.

Weight beaker containing insol. matter — Weight beaker =
Weight residue of pentane-insol. matter (V)

Combined weight of pentane-insol. matter from the weathered
specimen = IV + V (VI)

A control test is run to ascertain the pentane-insoluble matter in the unexposed asphalt. From this, the weight of pentane-insoluble matter in the particular specimen is calculated as follows:

$$\begin{aligned} &\text{Per cent pentane-insol. matter in unexposed asphalt} \times I = \\ &\text{Weight pentane-insol. matter in specimen before ex-} \\ &\text{posure} \dots\dots\dots (\text{VII}) \end{aligned}$$

Knowing the content of pentane-insoluble matter in the specimen before and after exposure, the increase can be calculated as follows:

$$\begin{aligned} &\text{Increase in pentane-insol. matter due to the weathering proc-} \\ &\text{ess} = \text{VI} - \text{VII} \dots\dots\dots (\text{VIII}) \end{aligned}$$

$$\begin{aligned} &\text{Loss in weight of asphalt during exposure} = \\ &\text{I} - \text{II} - \text{III} \dots\dots\dots (\text{IX}) \end{aligned}$$

This loss in weight of asphalt is considered as pentane-insoluble matter, and hence the total per cent increase in pentane-insoluble matter is calculated as follows:

$$\frac{\text{VIII} + \text{IX}}{\text{Weight oily constituents}} \times 100 \dots\dots\dots (\text{X})$$

NOTE.—The “Weight oily constituents” in unexposed specimen = per cent oily constituents in unexposed asphalt $\times I$.

Construct a “weathering-graph” by plotting the total per cent increase in pentane-insoluble matter (X), the average of duplicate determinations, on the ordinate, and the corresponding number of cycles (time) on the abscissa. Draw a curve and designate the appearance of surface cracks in the asphalt by one arrow, and the exposure of the aluminum by two arrows.

TEMPERATURE CONVERSION TABLE

FAHRENHEIT TO CENTIGRADE

F.°	0	10	20	30	40	50	60	70	80	90	Fractional Parts
	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	C.°	
0	-17.7	-12.2	-6.6	-1.1	+4.4	+10.0	+15.5	+21.1	+26.6	+32.2	
100	37.7	43.3	48.8	54.4	60.0	65.5	71.1	76.6	82.2	87.7	
200	93.3	98.8	104.4	110.0	115.5	121.1	126.6	132.2	137.7	143.3	
300	148.8	154.4	160.0	165.5	171.1	176.6	182.2	187.7	193.3	198.8	1 0.5
400	204.4	210.0	215.5	221.1	226.6	232.2	237.7	243.3	248.8	254.4	2 1.1
500	260.0	265.5	271.1	276.6	282.2	287.7	293.3	298.8	304.4	310.0	3 1.6
600	315.5	321.1	326.6	332.2	337.7	343.3	348.8	354.4	360.0	365.5	4 2.2
700	371.1	376.6	382.2	387.7	393.3	398.8	404.4	410.0	415.5	421.1	5 2.7
800	426.6	432.2	437.7	443.3	448.8	454.4	460.0	465.5	471.1	476.6	6 3.3
900	482.2	487.7	493.3	498.8	504.4	510.0	515.5	521.1	526.6	532.2	7 3.8
1000	537.7	543.3	548.8	554.4	560.0	565.5	571.1	576.6	582.2	587.7	8 4.4
											9 5.0

CENTIGRADE TO FAHRENHEIT

C.°	0	10	20	30	40	50	60	70	80	90	Fractional Parts
	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°	F.°	
-0	+32	+14	-4	-22	-40	-58	-76	-94	-112	-130	
+0	32	50	68	86	104	122	140	158	176	194	
100	212	230	248	266	284	302	320	338	356	374	
200	392	410	428	446	464	482	500	518	536	554	1 1.8
300	572	590	608	626	644	662	680	698	716	734	2 3.6
400	752	770	788	806	824	842	860	878	896	914	3 5.4
500	932	950	968	986	1004	1022	1040	1058	1076	1094	4 7.2
600	1112	1130	1148	1166	1184	1202	1220	1238	1256	1274	5 9.0
700	1292	1310	1328	1346	1364	1382	1400	1418	1436	1454	6 10.8
800	1472	1490	1508	1526	1544	1562	1580	1598	1616	1634	7 12.6
900	1652	1670	1688	1706	1724	1742	1760	1778	1796	1814	8 14.4
1000	1832	1850	1868	1886	1904	1922	1940	1958	1976	1994	9 16.2

Black figures indicate recurring decimals

Examples: 567° F. = 293.33 + 3.988 = 297.22° C.; - 85° C. = - 112 - 9.0 = - 121° F.



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CHAPTER VII

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CHAPTER X

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CHAPTER XXIV

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- p. 546 (60) U. S. Pats. 56363 of Jul. 17, 1866 to F. M. Buell; 58975 of Oct. 23, 1866 to Franklin Barse and G. E. Hopkins; 59551 of Nov. 13, 1866 to W. H. H. Burnham; 70478 of

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Nov. 5, 1867 to G. O. Smith and J. H. Smith; 73645 of Jan. 21, 1868 to S. B. Pierce and Pembroke Pierce; 77177 of Apr. 28, 1868 to Charles de Hass; 85453 of Dec. 29, 1868 to John Kivett and George Kivett; 86355 of Feb. 2, 1869 to J. W. Brown; 103581 of May 31, 1870 to E. J. De Smedt; 107756 of Sep. 27, 1870 to Charles Burgess; 113628 of Apr. 11, 1871 to John Cipperley; 124117 of Feb. 27, 1872 to D. G. Conger; 126026 of Apr. 23, 1872 to D. G. Conger; 142595 of Sep. 9, 1873 to Albert Thiele; 159629 of Feb. 9, 1875 to A. N. Atwood; 181528 of Aug. 29, 1876 to C. L. Fowler; 415864 of Nov. 26, 1889 to S. H. Gilson; 692627 of Feb. 4, 1902 to B. B. Clawson; 777173 of Dec. 13, 1904 to E. I. Allison; *Can. Pats.* 1808 of Nov. 21, 1872 to Elias Burnham; 2725 of Sep. 11, 1873 to Elias Burnham; 2726 of Sep. 12, 1873 to Elias Burnham; *Brit. Pats.* of 1871 (Apr. 19), 1035 to W. E. Newton; of 1872 (Aug. 22), 2491 to C. F. Seville; of 1898 (Nov. 14), 23981 to J. H. W. Stringfellow; of 1903 (Oct. 15), 22216 to A. E. Tanner and E. A. Claremont; of 1910 (Apr. 23), 9933 to R. C. Sharp; 369353 of Feb. 21, 1931 to F. W. Valle-Jones and C. J. W. Cloke; 252802 of Mar. 7, 1925 to J. A. W. Pine.

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p. 546 (62) *Brit. Pat.* of 1852 (Nov. 12), 719 to Charles Fox.

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p. 546 (73) *Brit. Pat.* 496952 of Dec. 3, 1938 to J. P. Spratling and P. O. Robinson.

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p. 547 (95) *Ger. Pat.* 107947 of Feb. 15, 1899 to Otto Pötzsch.

p. 547 (96) U. S. Pat. 84120 of Nov. 17, 1868 to Dwight Hitchcock.

p. 547 (97) U. S. Pats. 103095 of May 4, 1870 to G. H. Smith; 572645 of Dec. 8, 1896 to St. Vrain le Sieur; 1418905 of Jun. 6, 1922 to K. L. Binkley; *Ger. Pat.* 64680 of Sep. 23, 1891 to Bruno Roedelius.

p. 547 (98) U. S. Pat. 56818 of Jul. 31, 1866 to R. B. Smith.

p. 547 (99) U. S. Pat. 40650 of Nov. 17, 1863 to Abraham Straub.

p. 547 (100) *Ger. Pat.* 688492 of Feb. 1, 1940 to Sixten Magnus Hjelte.

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p. 547 (102) *Ger. Pats.* 95884 of Jun. 24, 1896 to Lebach & Co.; 680831 of Jun. 27, 1936 to Oskar Arendt.

p. 547 (103) U. S. Pat. 1774608 of Sep. 2, 1930 to H. L. Mead.

p. 547 (104) U. S. Pats. 52973 of Mar. 6, 1866 to Edward Curtis and Andrew Crozier; 59713 of Nov. 3, 1866 to Louis de l'Homme and Angelo Lazzaro; 95071 of Sep. 21, 1869 to Giacinto Bartolmei; *French Pat.* 813075 of May 25, 1937 to Charles Prochaska.

p. 547 (105) U. S. Pats. 1220682 of Mar. 27, 1917 to M. A. Popkess; 1364622 of Jan. 4, 1921 to J. R. Draney and L. M. Law; *Can. Pat.* 210186 of Apr. 5, 1921 to Bitoslag Paving Co.; *Brit. Pat.* 169079 of Jul. 30, 1920 to W. J. Mellersh-Jackson.

p. 547 (106) U. S. Pats. 29722 of Aug. 21, 1860 to George Scrimshaw; 34543 of Feb. 25, 1862 to W. H. White; 34653 of Mar. 11, 1862 to Zadok Street; 42589 of May 3, 1864 to H.

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Myers; 65660 of Jun. 11, 1867 to W. P. Ford and A. A. Moore; 69738 of Jul. 20, 1867 to Russell Fisk; 67998 of Aug. 20, 1867 to A. H. Mott, Daniel Winer and Lawrence Brink; 74963 of Feb. 25, 1868 to W. H. White; 87007 of Feb. 16, 1869 to T. Smith; 89186 of Apr. 20, 1869 to C. G. Von Tagen; 92390 of Jul. 6, 1869 to H. B. Steele; 104551 of Jun. 21, 1870 to J. W. Brown; 108693 of Oct. 25, 1870 to G. H. S. Duffus; 121118 of Nov. 21, 1871 to James McKenzie and J. M. Stebbins; 139848 of Jun. 17, 1873 to N. B. Abbott; 152503 of Jun. 30, 1874 to John McDerby and A. G. Stevens; 154778 of Sep. 8, 1874 to N. B. Abbott and J. P. Cranford; 161550 of Mar. 30, 1875 to J. P. Richardson; 162503 of Apr. 27, 1875 to Basile St. Jean; 1051769 of Jan. 28, 1913 to F. M. Ruschhaupt; 1237921 of Aug. 21, 1917 to E. J. Lovegrove and N. G. Crompton; 1352931 of Sep. 14, 1920 to Oscar Altpeter; 1369911 of Mar. 1, 1921 to T. H. Brown and Haughton Brown; 1370990 of Mar. 8, 1921 to B. W. O'Connell and Dennis O'Connell; *Can. Pat.* 8418 of Feb. 12, 1878 to John Brokenshire; *Brit. Pats.* of 1840 (Feb. 22), 8391 to Thomas Kerr; of 1869 (Dec. 18), 3672 to D. W. Bailey; of 1871 (May 16), 1315 to D. O. Macomber; of 1872 (Apr. 2), 966 to J. R. Croskey; of 1875 (Jun. 9), 4267 to Alexander Wilkinson; 361564 of Oct. 30, 1930 to Rudolf Traut.

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p. 547 (110) U. S. Pats. 46975 of Mar. 21, 1865 to C. G. Reinhold; 74606 of Feb. 18, 1868 to H. K. Schauk; 80084 of Jul. 21, 1868 to J. A. Moore; 169385 of Nov. 2, 1875 to L. P. Teed; 686191 of Nov. 5, 1901 to W. H. Bache; 853117 of May 7, 1907 to Clifford Richardson and C. N. Forrest; *Brit. Pats.* of 1884 (May 28), 8332 to Jules Hautrive; of 1887 (Sep. 17), 12632 to J. C. Lyman; of 1891 (Jun. 3), 9434 to John Menzies; *Ger. Pats.* 83096 of Jan. 16, 1894 to Carl Jost; 183552 of Nov. 18, 1904 to Hans Christen; *Austrian Pat.* 41300 of Mar. 10, 1910 to Emil Kuznitsky; *Japanese Pat.* 35965 of Mar. 11, 1920 to Y. Sakakibara.

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- p. 547 (118) Ger. Pat. 368236 of Dec. 24, 1920 to Lack- und Farbenfabriken Max Rogler.
- p. 547 (119) "New Packing Material (Sinterite) for Socket Joints," by Hans Vogt, *Gas- und Wasserfach*, 79, 592 (1936); U. S. Pats. 19627 of Mar. 16, 1858 to W. T. de Golyer; 47416 of Apr. 25, 1865 to Nicolaus Groh; 107232 of Sep. 13, 1870 to J. V. Douglass; 1465107 of Aug. 14, 1923 to Katharina Wickel; Can. Pat. 80476 of Apr. 21, 1903 to W. S. Wilkinson; Brit. Pats. of 1895 (Dec. 20), 24479 to R. D. Upham; of 1898 (Oct. 11), 21346 to Clemens Dörr; of 1913 (Nov. 14), 26173 to J. H. Francis; 183143 of Jul. 17, 1922 to Katharina Wickel; 435732 of Sep. 26, 1935 to N. V. de Bataafsche Petroleum Maatschappij; Ger. Pat. Appl. T-38898 of May 21, 1931 to Ernst Täuber.
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- p. 547 (121) Brit. Pat. of 1912 (Feb. 19), 4148 to J. Radcliffe.
- p. 547 (122) U. S. Pat. 69618 of Oct. 8, 1867 to A. T. Boon and Joseph Stafford.
- p. 547 (123) Brit. Pat. 424494 of Feb. 14, 1935 to Maurice Ernotte.
- p. 547 (124) U. S. Pats. 226547 of Apr. 13, 1880 to J. L. Pope; 452182 of May 12, 1891 to F. C. Goodall; 460249 of Sep. 29, 1891 to R. F. Flynn; 674219 of May 14, 1901 to J. A. Scharwath; 1796474 of Mar. 17, 1931 to G. A. Osterday; Can. Pats. 364137 of Feb. 16, 1937 to Barrett Co.; 387552 of Mar. 19, 1940 to Patent and Licensing Corp.; Brit. Pats. of 1844 (Nov. 9), 10387 to William Prosser, Jr.; of 1891 (Jun. 3), 9434 to John Menzies; French Pat. 762692 of Apr. 16, 1934 to André Léauté.
- p. 547 (125) U. S. Pats. 99267 of Jan. 25, 1870 to P. Werni; 102097 of Apr. 19, 1870 to N. H. Downs.
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- p. 547 (128) "Ageing of Coal-tar Coatings," by André Léauté, *Compt. rend.*, 197, 751, (1933); "Mélanges Goudron-Bitume," by S. L. Rashkovich, 3rd Congrès Belge de la Route, Brussels (1935); "Essais sur les Goudrons-fillers," by F. Campus and E. Dantinne, 3rd Congrès Belge de la Route, Brussels (1935); U. S. Pats. 62409 of Feb. 26, 1867 to David Green; 120236 of Oct. 24, 1871 to S. B. Brittain; 401014 of Apr. 9, 1889 to Alfonso de Fignanière; 565675 of Aug. 11, 1896 to E. T. Dumble; 1961678 of Jun. 5, 1934 to J. T. Sweeney; 2102480 of Dec. 14, 1937 to H. P. K. T. Nielsen; 2113794 of Apr. 12, 1938 to P. M. A. Léauté; Brit. Pats. of 1840 (Feb. 22), 8391 to Thomas Kerr; of 1875 (Jun. 9), 4267 to Alexander Wilkinson; of 1908 (Mar. 4), 4947 to V. A. Noodt and G. Götsche; 316897 of Mar. 3, 1928 to South Metropolitan Gas Co.; 329569 of Jul. 23, 1929 to South Metropolitan Gas Co.; 330440 of Jul. 23, 1929 to South Metropolitan Gas Co.; 334336 of Jul. 23, 1929 to South Metropolitan Gas Co.; 335668 of Jul. 23, 1929 to South Metropolitan Gas Co.; 341901 of Jul. 23, 1929 to South Metropolitan Gas Co.; 431474 of Jul. 8, 1935 to Soc. de Recherches et de Perfectionnements Industriels; Ger. Pat. Design 1277629 of Sep. 7, 1933 to Baeumer & Loesch; French Pat. Addition 48323 (809506) of Dec. 27, 1937 to Soc. anon. Salviam.
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- p. 548 (130) Ger. Pat. 598470 of Jan. 3, 1931 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger, A.-G.
- p. 548 (131) "Plastic Masses from Coal-tar Pitches," by H. Walter, *Korrosion u. Metallschutz*, 17, 351 (1941); U. S. Pats. 24105 of May 24, 1859 to J. M. Day and E. H. A. Oakley; 169385 of Nov. 2, 1875 to L. P. Teed; 425412 of Apr. 15, 1890 to I. T. Dyer; 727506 and 727507 of May 5, 1903 to F. J. Warren; 1864971 of Jun. 28, 1932 to J. H. Young and P. W. Jenkins; Can. Pat. 327880 of Nov. 22, 1932 to H. H. Robertson Co.; Brit. Pats. of 1856 (Aug. 25), 1976 to M. A. F. Mennons; of 1902 (Apr. 22), 9322 to H. H. Lake; 392372 of Jun. 28, 1932 to J. H. Young and P. W. Jenkins.
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CHAPTER XXV

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p. 560 (67) U. S. Pats. 992313 of May 16, 1911 to L. S. Van Westrum; 1472716 of Oct. 30, 1923 to W. P. Davey; 1640544 of Aug. 30, 1927 to W. T. Headley; 2232977 of Feb. 25, 1941 to C. H. Schuh; Can. Pats. 276480 of Dec. 20, 1927 to Mineral A.-G.; 411623 of Apr. 6, 1943 to Carbide & Carbon Chemicals Corp.; Brit. Pats. 233371 of Nov. 8, 1923 to L. S. Van Westrum; 269975 of Jan. 27, 1926 to L. S. Van Westrum; 276543 of Dec. 3, 1926 to Mineral A.-G.; 528308 of Oct. 25, 1940 to Bakelite Building Products Co.; Ger. Pats. 295893 of Jan. 19, 1910 to W. H. Elmenhorst; 378385 of Aug. 16, 1922 to Ernst Stern; 632300 of Jan. 10, 1933 to R. O. Bratke; French Pats. 611479 of Feb. 19, 1926 to Union française de Crédit; Addition 32974 (616407) of Jan. 24, 1927 to Soc. des Etablissements A. Lendormy; 706705 of Nov. 29, 1930 to Rütgerswerke A.-G.; Australian Pat. 1928/13805 of Jun. 12 to C. A. Hock; Czechoslovakian Pat. 30958 of Nov. 26, 1927 to Schottola & Co.; Hungarian Pat. 102050 of Feb. 10, 1930 to O. Janáček; Italian Pat. 274043 of Nov. 9, 1928 to G. Guadagni; Russian Pat. 47766 of Jul. 31, 1936 to B. A. Dogadkin and M. S. Lawrenenko.

p. 560 (68) U. S. Pat. 1706590 of Mar. 26, 1929 to Hermann Plauson; Swiss Pat. 122056 of Feb. 12, 1926 to Mineral A.-G.

p. 560 (69) U. S. Pats. 683375 of Oct. 1, 1901 to Sam Dyson & John Gaskell; 978307 of Dec. 13, 1910 to Samuel Knopf; 998691 of Jul. 25, 1911 to H. R. Kasson and S. S. Saxton; 1266956 of May 21, 1918 to Bruno Kniffier; 1542626 of Jun. 16, 1925 to H. A. Mackay; 1665105 of Apr. 3, 1928 to Robert Mezger; 1861398 of May 31, 1932 to R. Lant; 1861826 of Jun. 7, 1932 to L. G. Thompson; 1973599 of Sep. 11, 1934 to C. M. Baskin; 2003860 and 2003861 of Jun. 4, 1935 to K. E. McConaughay; 2087400 and 2087401 of Jul. 20, 1937 to W. F. Fair, Jr.; Can. Pats. 236318 of Dec. 11, 1923 to H. A. Mackay; 299042 of Apr. 8, 1930 to J. A. Montgomerie; 307169 of Dec. 30, 1930 to F. F. Lindstaedt; Brit. Pats. 202021 of Sep. 5, 1922 to H. A. Mackay; 202230 and 202231 of Sep. 5, 1922 to H. A. Mackay; 323633 of Jan. 23, 1924 to H. A. Mackay; 238967 of May 31, 1924 to G. S. Hay; 258870 of Sep. 20, 1926 to E. B. Hack; 305716 of Nov. 11, 1927 to J. A. Montgomerie; 311882 of Mar. 29, 1928 to F. W. Hammond; 319663 of Sep. 18, 1929 to C. G. J. Lefebvre and E. E. F. Berger; 333496 of Feb. 2, 1929 to Paul Lechler; 350040 of Mar. 11, 1930 to Patent and Licensing Corp.; 431642 of Dec. 6, 1933 to O. Bratke; 528308 of Oct. 25, 1940 to Bakelite Building Products Co.; 540831 of Oct. 31, 1941 to Bakelite Building Products Co.; Ger. Pats. 470306 of May 5, 1923 to Asphalt Cold Mix, Ltd.; 513846 of May 15, 1925 to F. Lydtin; 551665 of Feb. 6, 1926 to F. Lydtin; 553245 of Feb. 3, 1929 to Paul Lechler; 653929 of Jun. 25, 1931 to Colas Flintkote Ltd.; 654754 of Apr. 26, 1936 to Vereinigte Asphalt- und Teerprodukten-Fabriken G.m.b.H.; 674899 of Apr. 25, 1939 to Colas Kaltasphalt G.m.b.H.; French Pats. 564943 of Apr. 10, 1923 to Asphalt Cold Mix, Ltd.; Addition 36752 (564943) of Mar. 23, 1929 to Asphalt Cold Mix, Ltd.; 636207 of Oct. 15, 1926 to Soc. anom. La Trinidad; 744008 of Apr. 11, 1933 to Bitumen Investments Ltd.; 775229 of Dec. 21, 1934 to Alfred Tabary; 784724 of Jul. 23, 1935 to N. V. de Bataafsche Petroleum Maatschappij; Australian Pats. 1927/6847 of Apr. 11 to Asphalt Cold Mix, Ltd.; 1927/9126 of Jan. 30 to W. D. Summersfield; 1937/101176 of Jun. 10 to P. S. Robinson and J. D. Sutherland; Dutch Pat. 27610 of Sep. 15, 1932 to I. G. Farbenindustrie A.-G.; Swedish Pat. 57425 of Apr. 19, 1923 to Asphalt Cold Mix, Ltd.

p. 560 (70) U. S. Pats. 1547165 of Jul. 28, 1925 to G. T. Court and W. Karrer; 1804124 of May 5, 1931 to G. L. Southard; 1931072 of Oct. 17, 1933 to D. McK. Hepburn; 2285579 of

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Jun. 9, 1942 to Erich Gröner; *Can. Pat.* 300450 of May 20, 1930 to F. W. Atack; *Brit. Pat.* 319101 of Jul. 25, 1928 to Hermann Plauson; *Ger. Pats.* 343575 of Feb. 28, 1918 to K. Winkler & Co.; 363246 of Jun. 26, 1918 to L. S. Van Westrum; 407199 of Apr. 26, 1924 to Paul Lechler; *French Pat.* Addition 33627 (630168) of Mar. 4, 1927 to Henry Basset and Victor Szidon; *Indian Pat.* 19136 of Aug. 15, 1932 to McLeod & Co.

p. 560 (71) *U. S. Pats.* 1127831 of Feb. 9, 1915 to L. S. Van Westrum; 1259223 of Mar. 12, 1918 to W. M. Fraser; 1432742 of Oct. 24, 1922 to F. J. Commis; 1621483 of Mar. 15, 1927 to Howard Dimmig; 1957031 of May 1, 1934 to D. N. Myers; 1988336 of Jan. 15, 1935 to J. C. Roediger; 2027404 of Jan. 14, 1936 to J. B. Small; 2155141 of Apr. 18, 1939 to Cornelius Maters and M. J. Riemersma; 2199206 of Apr. 30, 1940 to Cornelius Maters and M. J. Riemersma; 2326610 of Aug. 10, 1943 to J. N. Borglin; *Can. Pats.* 229000 of Feb. 27, 1923 to F. J. Commis; 333862 of Dec. 17, 1930 to Bennett Inc.; *Brit. Pats.* 139491 and 139492 of Feb. 25, 1920 to W. M. Fraser; 252260 of Feb. 24, 1925 to G. S. Hay; 252449 of Nov. 27, 1924 to W. E. Billingshame; 406260 of May 18, 1932 to M. Crowne; *Ger. Pats.* 352357 of Sep. 1, 1918 to Carl Suchy and Rudolf Luszak; 392337 of Feb. 19, 1921 to Plauson's Forschungsinstitut G.m.b.H.; 446162 of Jun. 24, 1927 to Niels Bendixen, G. Buchner and A. Uhele; 549263 of Mar. 5, 1929 to Gesellschaft für Teerverwertung m.b.H. and Fritz Kraft; *French Pats.* 711782 of Nov. 13, 1931 to Imperial Chemical Industries Ltd.; 746754 of Jun. 6, 1933 to Sociedad Anonima Papeteries Navarre; 807958 of Jan. 26, 1937 to Thermal Industrial & Chem. Research Co. Ltd.; *Australian Pats.* 1930/28301 of Aug. 7 to S. K. Jones; 1931/2185 of May 20 to M. Crowne; *Austrian Pat.* 85603 of Sep. 26, 1921 to Carl Suchy and Rudolf Luszak; *Czechoslovakian Pat.* 48191 of Jan. 31, 1939 to B. Jerie; *Dutch Pat.* 20949 of Jun. 13, 1934 to Standard-Vacuum Oil Co.; *Indian Pat.* 20829 of May 2, 1934 to McLeod & Co.; *Italian Pats.* 276503 of Feb. 18, 1929 to A. F. Malchow A.-G.; 276770 of Feb. 13, 1929 to Vereinigte Dachpappen-Fabriken A.-G.; 276976 of Feb. 13, 1929 to Vereinigte Dachpappen A.-G.; *Swiss Pat.* 120508 of Feb. 1, 1926 to Asphalt Cold Mix, Ltd.

p. 560 (72) *Ger. Pats.* 571488 of Feb. 8, 1931 to Main-Gaswerke A.-G., Gas Passage and H. W. Hoelzer; 574528 of Feb. 20, 1932 to Main-Gaswerke A.-G. and H. W. Hoelzer.

p. 560 (73) *U. S. Pat.* 2198777 of Apr. 30, 1940 to Cornelius Maters and M. J. Riemersma; *Can. Pat.* 365239 of Apr. 6, 1937 to Flintkote Co.; *Brit. Pat.* 441782 of Aug. 2, 1934 to Colas Products, Ltd.; *Ger. Pat.* 648046 of Aug. 2, 1935 to N. V. de Bataafsche Petroleum Maatschappij; *French Pats.* 623924 of Oct. 30, 1926 to Michel Trux; 792874 of Jan. 11, 1936 to N. V. de Bataafsche Petroleum Maatschappij; *Australian Pat.* 1936/23721 of Mar. 26 to Asphalt Cold Mix, Ltd.

p. 560 (74) *U. S. Pats.* 2051409 and 2051410 of Aug. 18, 1936 to J. A. Kenney; 2217119 of Oct. 8, 1940 to E. G. Kerr; *Can. Pat.* 392289 of Nov. 5, 1940 to Barrett Co.; *Brit. Pats.* 312372 of Nov. 18, 1927 to G. Plauson; 438162 of Nov. 20, 1934 to J. R. Geigy A.-G.; *Ger. Pats.* 169493 of Feb. 1, 1907 to W. Spalteholz; 301927 of Dec. 19, 1916 to Georg Muth; 302632 of Dec. 5, 1916 to Georg Muth; 305271 of Jun. 21, 1917 to Georg Muth; 316345 of Jun. 6, 1916 to Georg Muth; 316617 of Apr. 23, 1919 to Georg Muth; 593184 of Mar. 12, 1929 to Gesellschaft für Teerstrassenbau m.b.H.; 602165 of Nov. 21, 1933 to J. R. Geigy A.-G.; 648046 of Aug. 2, 1935 to N. V. de Bataafsche Petroleum Maatschappij; 669152 of Dec. 17, 1938 to L. A. Svensson; *Appl. G-88052* of May 11, 1934 to J. R. Geigy A.-G.; *French Pat.* 781331 of May 13, 1935 to J. R. Geigy A.-G.; *Swedish Pat.* 95304 of Apr. 5, 1939 to L. A. Svensson; *Swiss Pat.* 180680 of Sep. 11, 1934 to J. R. Geigy A.-G.

p. 560 (75) *U. S. Pats.* 1565125 of Dec. 8, 1925 to L. S. Van Westrum; 2163445 of Jun. 20, 1939 to L. G. Gabriel and J. F. T. Blott; 2350548 of Jun. 6, 1944 to W. W. De Laney; *Brit. Pats.* 275364 of May 2, 1929 to L. S. Van Westrum; 466510 of Nov. 29, 1935 to E. E. Mayer; *Ger. Pats.* 52129 of May 8, 1889 to W. Dammann; 459655 of Feb. 1, 1925 to L. S. Van Westrum; 509574 of Jul. 7, 1928 to Josef Klein; 565057 of Oct. 12, 1932 to Deutsche Hydrierwerke A.-G.; *French Pat.* 608123 of Dec. 21, 1925 to Soc. Rol. Lister et Cie.

p. 560 (76) *Brit. Pat.* 202235 of Sep. 5, 1922 to H. A. Mackay; *French Pats.* 781161 of May 10, 1935 to Société Paix et Cie.; 833170 of Oct. 13, 1938 to Soc. chimique et routiere de la gironde; *Austrian Pat.* 116850 of May 4, 1923 to Asphalt Cold Mix, Ltd.

- p. 560 (77) Brit. Pat. 233430 of Feb. 8, 1924 to H. A. Mackay; French Pat. 752178 of Mar. 9, 1933 to Fernando Elosegui; Swiss Pat. 164109 of Dec. 1, 1933 to Hermann Plauson.
- p. 560 (78) U. S. Pat. 2331022 of Oct. 5, 1943 to R. J. Garofalo and F. S. Scott; Brit. Pat. 320357 of Jul. 9, 1928 to Josef Klein; Ger. Pats. 122451 of Jun. 6, 1899 to F. Boleg; 129480 of May 1, 1900 to F. Boleg; 658439 of Mar. 13, 1935 to Erwin Dornig.
- p. 560 (79) U. S. Pat. 1665881 of Apr. 10, 1928 to Eugen Hutzenlaub; Ger. Pats. 309680 of Jan. 4, 1918 to W. Schmidt and E. Heuser; 631781 of Apr. 6, 1933 to Eugène Rouault; Brit. Pat. 431642 of Aug. 8, 1935 to O. Bratke; French Pat. 832683 of Sep. 30, 1938 to Standard française des pétroles.
- p. 560 (80) U. S. Pat. 2080689 of May 18, 1937 to U. B. Bray and L. B. Beckwith; French Pats. 763289 of Apr. 26, 1934 to O. Bratke; 767793 of Jul. 24, 1934 to I. G. Farbenindustrie A.-G.; Norwegian Pat. 41999 of Mar. 12, 1925 to P. S. Kaug.
- p. 560 (81) U. S. Pat. 2256886 of Sep. 23, 1941 to W. D. Buckley; Brit. Pat. 251148 of Jul. 24, 1925 to B. C. Sellers; Ger. Pat. 566670 of Nov. 15, 1929 to Chem. Fabrik Flörsheim, Dr. H. Noerdlinger A.-G.; French Pats. 810310 of Mar. 19, 1937 to Élie Sansoube; 820913 of Nov. 22, 1937 to Standard française des pétroles.
- p. 560 (82) Ger. Pat. 338955 of Jan. 4, 1920 to Rütgerswerke A.-G.
- p. 560 (83) Brit. Pat. 263307 of Jan. 5, 1926 to C. H. Thompson and W. J. McGivern; French Pat. 630168 of Mar. 4, 1927 to Henry Basset and Victor Szidon.
- p. 560 (84) Can. Pat. 341465 of May 8, 1934 to Bennett, Inc.; Brit. Pats. 308051 of Feb. 16, 1928 to J. A. Montgomerie; 315057 of May 23, 1928 to C. H. Thompson and W. J. McGivern; French Pat. 613084 of Jul. 9, 1925 to Georges Baume, Pierre Chambige and de Boutier.
- p. 560 (85) U. S. Pats. 1940431 and 1940432 of Dec. 19, 1933 to O. F. Nietzsche; 2321240 of Jun. 8, 1943 to R. B. Porter, Jr.; Can. Pat. 323861 of Jul. 5, 1932 to Bennett, Inc.; Ger. Pats. 308442 of Jan. 25, 1917 to St. Rochus, G.m.b.H.; 357378 of Feb. 8, 1921 to Chemische Werkstätten; 398879 of Oct. 3, 1922 to Chemische Werkstätten; 409690 of Dec. 31, 1921 to Deutsche Erdöl A.-G. and W. R. Röderer; 514485 of Mar. 22, 1925 to Bitumuls Kaltasphalt A.-G.; 556250 of May 5, 1928 to Deutsche Erdöl A.-G.; 575104 of Dec. 21, 1926 to Heinrich Kretzer; 584695 of Oct. 14, 1928 to Heinrich Kretzer; Appl. J-43070 of Nov. 11, 1931 to I. G. Farbenindustrie A.-G.; French Pat. 684048 of Oct. 29, 1929 to Agasote Millboard Co.
- p. 560 (86) Brit. Pat. 387825 of Jan. 18, 1932 to H. A. Gill; Ger. Pat. 542605 of Dec. 10, 1926 to Gesellschaft für Teerstrassenbau, m.b.H.
- p. 560 (87) Brit. Pat. 251323 of Jan. 1, 1925 to G. S. Hay; Ger. Pats. 365160 of Mar. 25, 1919 to Peter Friesenhahn; 615563 of Jul. 8, 1935 to G. Kropfhammer; French Pats. 648138 of Feb. 3, 1928 to Meilach Melamid; 698554 of Jun. 20, 1930 to Hugo Novák; 717299 of Jan. 6, 1932 to I. G. Farbenindustrie A.-G.; Australian Pats. 1927/8551 of Jul. 29 to Hart & Co., Pty., Ltd.; 1930/28301 of Aug. 7 to Neuchatel Asphalt Co. Ltd.; Indian Pat. 20829 of May 2, 1934 to McLeod & Co.
- p. 560 (88) "New Commercial Emulsifying Agents," by Harry Bennett, *J. Ind. Eng. Chem.*, 22, 1255 (1930); Brit. Pat. 437674 of Nov. 4, 1935 to E. I. Dupont de Nemours & Co.; French Pat. 717390 of May 20, 1931 to Frédéric Steinfels (S.A.).
- p. 560 (89) U. S. Pat. 1988879 of Jan. 22, 1935 to H. M. Steininger; Brit. Pat. 381286 of Aug. 16, 1931 to N. V. Tot Voortzetting der Zaken van Pieter Schoen & Zoon.
- p. 560 (90) Brit. Pat. 455540 of Oct. 22, 1936 to H. C. Lundsgaard.
- p. 560 (91) French Pat. 740494 of Jan. 26, 1933 to I. G. Farbenindustrie A.-G.
- p. 560 (92) Swiss Pat. 151650 of Mar. 16, 1932 to Chemische Fabrik vorm. Sandoz.
- p. 560 (93) U. S. Pat. 2269529 of Jan. 13, 1942 to H. A. Goldsmith.
- p. 560 (94) U. S. Pat. 2052025 of Aug. 25, 1936 to B. R. Harris.
- p. 560 (95) Ger. Pats. 545763 of Mar. 10, 1929 to Th. Goldschmidt, A.-G.; 551403 of Aug. 28, 1927 to Th. Goldschmidt, A.-G.; 582106 of Aug. 9, 1933 to Th. Goldschmidt, A.-G.; 590165 of Feb. 15, 1928 to Th. Goldschmidt, A.-G.
- p. 560 (96) Brit. Pat. 393276 of Jun. 29, 1932 to Imperial Chemical Industries, Ltd.; French Pat. 746440 of May 29, 1932 to Imperial Chemical Industries, Ltd.
- p. 560 (97) U. S. Pats. 1938804 of Dec. 12, 1933 to Mendel Burak; 2114689 of Apr. 19,

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- 1938 to A. D. Smith; *Brit. Pats.* 333152 and 333153 of Feb. 4, 1929 to C. G. Fox; *Hungarian Pat.* 101458 of Feb. 10, 1930 to O. Janáček.
- p. 560 (98) *Swiss Pat.* 156724 of Nov. 1, 1932 to I. G. Farbenindustrie A.-G.
- p. 560 (99) *Ger. Pat.* 432942 of Dec. 11, 1923 to Ernst Schmidt.
- p. 560 (100) *Can. Pat.* 387403 of Mar. 12, 1940 to Carbide & Carbon Chemicals Ltd.
- p. 560 (101) *French Pat.* 745113 of May 5, 1933 to I. G. Farbenindustrie, A.-G.
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p. 561 (130) **U. S. Pats.** 867141 of Sep. 24, 1907 to O. A. H. H. Kösters; 884878 of Apr. 14, 1908 to J. P. Van Der Ploeg; 956009 of Apr. 26, 1910 to L. S. Van Westrum; 1778239 and 1778240 of Oct. 14, 1930 to W. J. Yates; 1854348 of Apr. 19, 1932 to Gustav Rodewald; 2049043 of Jul. 28, 1936 to H. S. Birkby; **Can. Pat.** 297097 of Dec. 28, 1930 to C. G. Merrell; **Brit. Pats.** of 1905 (Apr. 11), 7699 to J. P. Van der Ploeg; of 1905 (Jun. 2), 11620 to R. M. Hahn; 229361 of Sep. 19, 1923 to H. A. Mackay; 255044 of Jun. 22, 1926 to Paul Lechler; 294582 of Jul. 18, 1929 to Gesellschaft für Chemische Industrie; 300821 of Dec. 24, 1927 to J. A. Montgomerie; 332897 of Mar. 20, 1929 to C. G. Fox; 358202 of Oct. 29, 1931 to Gesellschaft für Chemische Industrie; 364104 of Jan. 28, 1932 to I. G. Farbenindustrie A.-G.; 371822 of Jun. 26, 1932 to I. G. Farbenindustrie A.-G.; 372005 of May 26, 1932 to I. G. Farbenindustrie A.-G.; 416658 of Dec. 14, 1933 to H. Hunsdiecker and E. Vogt; 418247 of Jul. 11, 1933 to Resinour Products & Chemical Co.; 450672 of Jul. 17, 1936 to J. W. Orelup; **Ger. Pats.** 191399 of Apr. 8, 1905 to J. P. Van Der Ploeg; 363374 of Jul. 8, 1921 to Sanitol-Werke, G.m.b.H.; 453465 of Dec. 6, 1927 to Montanwerke A.-G.; 549330 of Mar. 19, 1929 to Amber Size & Chemical Co., Ltd.; 552251 of Oct. 6, 1932 to Deutsche Hydrierwerke A.-G.; **French Pat.** 785006 of Jul. 31, 1935 to H. Th. Böhme A.-G.; **Swiss Pat.** 148719 of May 1, 1930 to Romag A.-G. für Rohmaterialien.

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p. 561 (132) "Types of Hydrocarbon-oil Emulsions," by W. Seifriz, *J. Phys. Chem.*, 29, 587, 595, 600, 746 and 838 (1925); "Casein and Its Industrial Applications," by Edwin Sutermeister and F. L. Browne, 2nd Edition; Reinhold Publishing Co., New York (1939); "Casein and Its Uses," by Hans Hadert, Nordemann Publishing Co., Inc., New York (1938). **U. S. Pats.** 786348 of Apr. 4, 1905 to L. A. Dreyfus; 788043 of Apr. 25, 1905 to F. X. Govers; 788857 of May 2, 1905 to G. A. Thubé and Louis Préaubert; 790821 of May 23, 1905 to H. V. Dunham; 812593 of Feb. 13, 1906 to Louis Préaubert and G. A. Thubé; 1010210 of Nov. 28, 1911 to C. L. V. Zimmer; 1357688 of Nov. 2, 1920 to Henry Chislet; 1549436 of May 15, 1925 to W. E. Billingham; 1700581 of Jan. 29, 1929 to W. E. Billingham; 1859517 of May 24, 1932 to Lester Kirschbraun; 2040115 of May 12, 1936 to V. E. Watts; 2074731 of Mar. 23, 1937 to C. L. McKesson; 2172392 of Sep. 12, 1939 to Otto Kress and C. E. Johnson; 2223642 of Mar. 11, 1941 to J. F. T. Blott and J. A. Rawlinson; 2267810 of Dec. 30, 1941 to U. B. Bray and L. B. Beckwith; **Can. Pats.** 229000 of Feb. 27, 1923 to F. J. Commis; 247347 of Mar. 3, 1925 to N. V. Koninklijke Stearin Kaarsenfabriek Gouda; 255518 of Nov. 17, 1925 to Asphalt Cold Mix, Ltd.; 2611175 of May 25, 1926 to Tarkold, Ltd.; 286384 of Jan. 15, 1929 to W. E. Billingham; 325450 of Aug. 30, 1932 to Miroslav Hubmajer; 330943 of Mar. 14, 1933 to International Bitumen Emulsions Corp.; 342801 of Jul. 3, 1934 to International Bitumen Emulsions Corp.; **Brit. Pats.** of 1903 (May 14), 11028 to Louis Préaubert and G. A. Thubé; of 1905 (May 5), 9422 to Louis Préaubert and G. A. Thubé; 155398 of Sep. 30, 1919 to E. Schou; 212248 of Feb. 28, 1924 to Thomson-Houston Co., Ltd.; 221380 of Nov. 7, 1923 to L. W. Low; 222602 of Nov. 19, 1923 to W. E. Billingham; 225587 of Jun. 6, 1923 to Knud Erslev; 230177 of Dec. 5, 1923 to H. A. Mackay; 251098 of Apr. 28, 1925 to W. J. McGivern, J. H. Foster and

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R. Swift; 252260 of Feb. 24, 1925 to G. S. Hay; 255074 of Jul. 7, 1926 to Georges Baume, Pierre Chambi and de Boutier; 255911 of Jul. 23, 1926 to W. E. Billingham; 258870 of Sep. 20, 1926 to E. B. Hack; 264955 of Nov. 10, 1925 to I. T. Jones; 286552 of Aug. 2, 1927 to N. V. Koninklijke Stearin Kaarsenfabriek Gouda; 301805 of Dec. 3, 1928 to N. V. de Bataafsche Petroleum Maatschappij; 308389 of Jan. 7, 1928 to T. M. Hickman; 333303 of May 14, 1929 to Jonathan Parker; 365586 of Aug. 15, 1930 to Ragnvald Hellerud; 374111 of Mar. 4, 1931 to J. A. Montgomerie; 393868 of Nov. 19, 1932 to C. G. Fox and S. Stockwell; 401131 of Nov. 9, 1933 to International Bitumen Emulsions Corp.; 418107 of Aug. 25, 1925 to Barnold Ltd.; 434109 and 434180 of Jun. 16, 1934 to Roberts & Smith; 462111 of Mar. 2, 1937 to J. A. Montgomerie and P. K. Archibald; 480097 of Feb. 14, 1938 to Institute of Paper Chemistry; **Ger. Pats.** 239828 of Nov. 25, 1911 to G. Nohl; 240482 of May 1, 1910 to A.-G. für Asphaltierung und Dachbedeckung vorm. Johannes Jeserich; 342639 of Dec. 24, 1920 to F. J. Commin; 405930 of Nov. 22, 1922 to Ernst Stern; 418107 of Oct. 23, 1924 to Tarkold, Ltd.; 514399 of Jul. 17, 1927 to Hermann Bollmann and Bruno Rewald; 516187 of Sep. 7, 1927 to Hermann Bollmann and Bruno Rewald; 516188 of Oct. 25, 1927 to Hermann Bollmann and Bruno Rewald; 516189 of Dec. 25, 1927 to Hermann Bollmann and Bruno Rewald; 547895 of Apr. 1, 1932 to C. A. Agthe; 584949 of Sep. 26, 1933 to Chem. Seifenfabrik vorm. A. Baumheier; 643350 of Aug. 28, 1931 to Chemische Fabrik Flörsheim, Dr. H. Noerdlinger A.-G.; **Appl. G-91234** of Sep. 9, 1935 to Th. Goldschmidt A.-G.; **French Pats.** 529295 of Dec. 31, 1920 to F. J. Commin; 586847 of Oct. 4, 1924 to W. E. Billingham; 588016 of Oct. 20, 1924 to Tarkold, Ltd.; 606535 of Nov. 11, 1925 to W. E. Billingham; 623777 of Oct. 28, 1926 to G. Murphy; Addition 41542 of Jan. 28, 1933 to I. G. Farbenindustrie A.-G.; 732463 of Feb. 29, 1932 to Bitumen Investments, Inc.; **Australian Pats.** 19922 of Oct. 6, 1924 to Asphalt Cold Mix Ltd.; 20327 of Oct. 31, 1924 to W. E. Billingham; 20442 of Nov. 7, 1924 to Tarkold Ltd.; **Austrian Pat.** 130450 of Jan. 1, 1928 to N. V. Vereengde Fabrieken van Stearine, Kaarsen en chemische Produkten; **Swiss Pat.** 112803 of Sep. 17, 1924 to Asphalt Cold Mix, Ltd.

p. 561 (133) **Brit. Pat.** 308339 of Jan. 7, 1928 to F. M. Hickman.

p. 561 (134) **U. S. Pat.** 2074731 of Mar. 23, 1937 to C. L. McKesson; **Can. Pat.** 379270 of Jan. 31, 1939 to International Bitumen Emulsions Ltd.; **Brit. Pat.** 475387 of Feb. 3, 1937 to International Bitumen Emulsions Ltd.; **French Pat.** 817510 of Sep. 4, 1937 to International Bitumen Emulsions Ltd.

p. 561 (135) **U. S. Pats.** 1466022 of Aug. 28, 1923 to Hatsugoro Kurata; 1855934 of Apr. 26, 1932 to H. P. Banks, Glenn Davidson, I. F. Laucks and H. F. Rippey; 2020662 of Nov. 12, 1935 to Albert Schwiager; 2336468 of Dec. 14, 1943 to W. G. Cole and W. D. Buckley; **Can. Pat.** 387131 of Feb. 27, 1940 to Institute of Paper Chemistry; **Brit. Pats.** 382432 of Nov. 2, 1931 to Hanseatische Mühlenwerke A.-G.; 505983 of May 17, 1939 to I. G. Farbenindustrie A.-G.; **Ger. Pats.** 239828 of Feb. 26, 1908 to G. Nohl; 522041 of Dec. 6, 1927 to Hanseatische Mühlenwerke, A.-G.; 557154 of Nov. 16, 1930 to I. G. Farbenindustrie A.-G.; 557288 of Aug. 17, 1930 to Gesellschaft für Teerverwertung m.b.H., Fritz Kraft and Theodor Heydecke; 594189 of Apr. 16, 1933 to Hanseatische Mühlenwerke A.-G.; 604328 of Dec. 2, 1932 to Deutsche Vitalit-Gesellschaft, m.b.H.; **French Pats.** 730961 of Feb. 3, 1932 to Gesellschaft für Teerverwertung, m.b.H.; 771284 of Oct. 4, 1934 to Hanseatische Mühlenwerke A.-G.

p. 561 (136) **U. S. Pat.** 1950272 of Mar. 6, 1934 to A. E. Schutte.

p. 561 (137) **U. S. Pats.** 1440355 of Dec. 26, 1922 to J. C. Morrell; 1755379 of Apr. 22, 1930 to H. W. Banks; 1960115 of May 22, 1934 to Z. C. Loebel; 1969659 of Aug. 7, 1934 to W. W. McLaurin; **Can. Pats.** 313274 of Jul. 14, 1931 to Flintkote Roads, Inc.; 333343 of Jun. 20, 1933 to W. W. McLaurin; **Brit. Pats.** 255074 of Jul. 7, 1926 to Georges Baume; 274142 of Jan. 15, 1926 to J. H. Disney and J. C. Kernot; 322792 and 322793 of Sep. 13, 1928 to Colas Products Ltd., L. G. Gabriel and J. F. Blott; 323060 of Oct. 6, 1928 to F. N. Nicholls; **Ger. Pats.** 127582 of Jun. 17, 1900 to Carl Baswitz; 312690 of May 10, 1912 to W. Plinatus; 421237 of May 3, 1924 to A. Knecht; **Austrian Pat.** 124728 of Oct. 10, 1931 to N. V. de Bataafsche Petroleum Maatschappij; **Norwegian Pat.** 45902 of Dec. 20, 1927 to M.I.D. Syndicate, Ltd.

p. 561 (138) U. S. Pat. 1647805 of Nov. 1, 1927 to John McGavack; Brit. Pats. 221466 of Feb. 27, 1924 to Douglas Pectin Corp.; 276878 of Oct. 5, 1926 to California Fruit Growers' Exchange; Ger. Pats. 384772 of Apr. 11, 1917 to Berlin Dextrinfabrik, Otto Kutzner; 560259 of Feb. 7, 1932 to Deutsche Pektingesellschaft m.b.H.

p. 561 (139) U. S. Pats. 834830 of Oct. 30, 1906 to Karl Mann; 943667 of Dec. 21, 1909 to Carleton Ellis; 1754535 of Apr. 15, 1930 to W. B. Wescott; 1831492 of Nov. 10, 1931 to E. A. Hauser; 1869526 of Aug. 2, 1932 to M. L. Tower, H. W. Dye and F. L. McDonough; Brit. Pats. 239120 of May 31, 1924 to G. S. Hay; 255456 of Jul. 13, 1926 to C. Glücksmann; 286527 of Jun. 15, 1927 to Johnson & Johnson; 337269 of Jan. 24, 1929 to K. D. P., Ltd.; 351242 of Jan. 26, 1930 to F. J. E. China and W. A. White; 405906 of Feb. 15, 1934 to W. H. Wilcken; 493898 of Dec. 13, 1937 (Addition to 430061) to Société de Recherches et de Perfectionnements Industriels; Ger. Pat. 127852 of Jan. 18, 1902 to Carl Baswitz; French Pats. 686821 of Dec. 18, 1929 to K. D. P., Ltd.; 760958 of Mar. 7, 1934 to W. H. Wilcken; Addition 49103 (773848) of Nov. 7, 1938 to Société de Recherches et de Perfectionnements Industriels.

p. 561 (140) French Pats. 633687 of Sep. 6, 1926 to P. Gloess and M. Marini; 684905 of Feb. 14, 1929 to P. Gloess.

p. 561 (141) U. S. Pats. 1653026 of Dec. 20, 1927 to F. C. Thornley, F. F. Tapping and Otto Reynard; 1814986 of Jul. 14, 1931 to M. J. Walsh; Can. Pats. 252659 of Aug. 11, 1925 to F. C. Thornley and F. F. Tapping; 338571 of Jan. 9, 1934 to G. S. Rutherford; Brit. Pats. 219348 of Jan. 26, 1923 to F. C. Thornley and F. F. Tapping; 294002 of Aug. 25, 1927 to General Rubber Co.; French Pats. Addition 34089 (578564) of Jun. 8, 1927 to P. Gloess; 583759 of Jul. 15, 1924 to F. C. Thornley; 618918 of Jul. 13, 1926 to Chem. Fabr. Dr. Joachim Wiernick.

p. 561 (142) U. S. Pats. 1582467 of Apr. 27, 1926 to G. S. Hay; 1738022 of Dec. 3, 1929 to J. P. Strasser; 1881729 of Oct. 11, 1932 to H. L. Levin; 2044570 of Jun. 16, 1936 to C. E. Hite; 2047258 of Jul. 14, 1936 to Maurice Ernotte; 2347678, 2347679 and 2347680 of May 2, 1944 to K. M. Gaver; Can. Pats. 264635 of Sep. 28, 1926 to Asphalt Cold Mix, Ltd.; 266901 of Dec. 21, 1926 to Asphalt Cold Mix, Ltd.; 349595 of Apr. 16, 1935 to Flintlock Co.; Brit. Pats. of 1893 (Nov. 11), 8196 to Henry Helbing; 243398 of May 31, 1924 to G. S. Hay; 248859 of Dec. 16, 1924 to G. S. Hay; 305742 of Nov. 26, 1927 to E. C. R. Marks; 307079 of Mar. 2, 1929 to Oranienburger Chemische Fabrik A.-G.; 344490 of Jul. 9, 1929 to Union Chimique Belge, S. A.; 346978 of Apr. 24, 1930 to Niels Bendixen and J. G. Y. D. Morgan; 419358 of Apr. 5, 1933 to Eugène Rouault; 504500 of May 6, 1938 to Standard Oil Development Co.; 509174 of Apr. 5, 1938 to Alfred Halward and Peter Murányi; Ger. Pats. 170133 of Jun. 3, 1904 to Karl Mann; 575922 of Oct. 9, 1927 to Ivo Dieglmayr; 585586 of Aug. 2, 1934 to Ivo Dieglmayr; 590958 of Apr. 10, 1932 to I. G. Farbenindustrie, A.-G.; 653929 of Dec. 10, 1937 to Colas Flintlock Ltd.; Appl. K-128183 of Dec. 13, 1932 to Rudolf Köhler and Hans Erbring; Appl. E-45516 of Apr. 17, 1934 to Maurice Ernotte; French Pats. 591040 of Dec. 27, 1924 to Asphalt Cold Mix, Ltd.; 748886 of Jul. 12, 1933 to Eugène Rouault; 836354 of Jan. 17, 1939 to Alfred Halward and Peter Murányi; Czechoslovakian Pat. 32911 of Aug. 18, 1925 to Asphalt Cold Mix, Ltd.; Danish Pat. 33864 of Mar. 14, 1924 to H. Lange; Japanese Pat. 91327 of May 9, 1931 to Keita Maki.

p. 561 (143) "Dust Preventatives and Road Binders," by Prévost Hubbard, pp. 109 et seq. John Wiley & Sons, New York (1910); U. S. Pats. 62810 of Mar. 12, 1867 to A. F. Boon; 349751 of Sep. 28, 1886 to A. H. Rowand and R. S. Hunzeker; 2136667 of Nov. 15, 1938 to E. J. Bert; Ger. Pat. 632015 of Jul. 1, 1936 to Maurice Ernotte; Australian Pat. 1926/359 of Feb. 2 to J. W. Pohlmann; Austrian Pat. 101032 of Mar. 27, 1923 to A. Neumann; Belgian Pats. 395761 of May 31, 1933 to Maurice Ernotte; 402135 of Apr. 30, 1934 to Maurice Ernotte; Japanese Pat. 129057 of Feb. 28, 1939 to Kozi Doi.

p. 561 (144) U. S. Pats. 1440356 of Dec. 26, 1922 to J. C. Morrell; 1832987 of Nov. 24, 1931 to Lester Kirschbraun; 1840157 of Jan. 5, 1932 to C. F. Cross and Alf Engelstad; 1878828 of Sep. 20, 1932 to C. F. Cross and Alf Engelstad; 1988543 of Jan. 22, 1935 to Karl Daimler; 2002505 of May 28, 1935 to A. W. Hixson and J. M. Fain; 2008978 of Jul. 23, 1935 to Karl Daimler; Can. Pats. 262783 of Jul. 20, 1926 to Asphalt Cold Mix, Ltd.; 288853 of Apr. 16,

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1929 to I. G. Farbenindustrie, A.-G.; *Brit. Pats.* 246907 of Nov. 3, 1924 to Asphalt Cold Mix, Ltd. and Frank Levy; 263520 of Sep. 21, 1925 to C. F. Cross and Alf Engelstad; 317496 of Mar. 16, 1928 to I. G. Farbenindustrie, A.-G.; 334426 of Oct. 20, 1928 to I. G. Farbenindustrie, A.-G.; 351904 of Dec. 15, 1928 to I. G. Farbenindustrie, A.-G.; 362467 of Jul. 5, 1930 to I. G. Farbenindustrie, A.-G.; 366158 of Nov. 7, 1930 to I. G. Farbenindustrie, A.-G.; 366174 of Nov. 17, 1930 to I. G. Farbenindustrie, A.-G.; 367782 of Nov. 18, 1930 to I. G. Farbenindustrie, A.-G.; 381933 of May 27, 1931 to I. G. Farbenindustrie, A.-G.; 430917 of Dec. 27, 1933 to W. W. Groves; 433550 of Feb. 16, 1934 to W. W. Groves; 441879 of Jul. 27, 1934 to W. W. Groves; *Ger. Pats.* 313339 of Mar. 17, 1914 to W. Riese; 352860 of May 6, 1922 to Farbwerke vorm. Meister Lucius & Brüning; 390434 of Mar. 28, 1922 to Plauson's Forschungsinstitut, G.m.b.H.; 461833 of Mar. 1, 1924 to C. F. Cross and Alf Engelstad; 562050 of Apr. 15, 1931 to I. G. Farbenindustrie, A.-G.; 591340 of Dec. 19, 1929 to Gesellschaft für Teerverwertung, m.b.H.; 627465 of Nov. 8, 1933 to I. G. Farbenindustrie, A.-G.; 629270 of Apr. 25, 1936 to I. G. Farbenindustrie, A.-G.; 653958 of Nov. 12, 1933 to I. G. Farbenindustrie A.-G.; *French Pats.* 559225 of Aug. 14, 1924 to Soc. de Recherches; 606418 of Nov. 3, 1925 to Asphalt Cold Mix, Ltd.; 705797 of Nov. 15, 1930 to I. G. Farbenindustrie, A.-G.; 707505 of Dec. 13, 1930 to I. G. Farbenindustrie, A.-G.; 714853 of Dec. 11, 1931 to Miroslav Hubmajer; 725296 of Jan. 7, 1931 to Soc. des Produits Chimiques et Matières Colorantes de Mulhouse; *Australian Pat.* 1929/21709 of Aug. 8 to Colitho Pavements Proprietary Ltd.; *Belgian Pat.* 350865 of Apr. 25, 1928 to I. G. Farbenindustrie A.-G.; *Jugoslavian Pat.* 6174 of Apr. 10, 1928 to I. G. Farbenindustrie A.-G.; *Norwegian Pat.* 58672 of Nov. 15, 1937 to Olav Ovregard; *Swiss Pat.* 138803 of Apr. 18, 1928 to I. G. Farbenindustrie A.-G.

p. 561 (145) *Brit. Pat.* of 1905 (Jun. 2), 11620 to R. M. Hahn.

p. 561 (146) *U. S. Pat.* 1997868 of Apr. 16, 1935 to H. L. Levin.

p. 561 (147) *French Pat.* 801034 of Jul. 25, 1936 to Chemische Forschungsgesellschaft, m.b.H.

p. 561 (148) *U. S. Pat.* 1973991 of Sep. 18, 1934 to C. L. McKesson, L. G. Thompson and W. D. Buckley.

p. 561 (149) *U. S. Pat.* 2005113 of Jun. 18, 1935 to Orvall Smiley; *Can. Pat.* 328800 of Dec. 27, 1932 to Orvall Smiley.

p. 561 (150) *U. S. Pats.* 1783366 of Dec. 2, 1930 to R. W. Lewis; 1957408 of May 1, 1934 to Eugen Hutzenlaub; *Can. Pat.* 297226 of Feb. 4, 1930 to J. A. Montgomerie; *Brit. Pats.* 321334 of Nov. 17, 1928 to J. A. Montgomerie; 351073 of May 9, 1930, to C. M. Cunynghame and C. W. Fulton; 428571 of Aug. 1, 1934 to G. M. Skinner, Ltd.; 436494 of Mar. 18, 1935 to F. V. Lister; *French Pats.* 832236 of Jan. 18, 1938 to Dimitrie Frunzetti; 793070 of Jun. 17, 1935 to Gustave Labourse; *Australian Pat.* 1933/13953 of Aug. 17 to G. M. Skinner.

p. 561 (151) *U. S. Pats.* 1738776 of Dec. 10, 1929 to Lester Kirschbraun; 2033657 of Mar. 10, 1936 to P. R. Smith; *Brit. Pat.* 436494 of Oct. 11, 1935 to F. V. Lister; *French Pat.* 802436 of Feb. 26, 1936 to F. V. Lister.

p. 561 (152) *U. S. Pat.* 1788706 of Jan. 13, 1931 to Roy Cross; *Brit. Pat.* 494380 of Oct. 25, 1938 to Dimitrie Frunzetti; *French Pat.* 832236 of Sep. 23, 1938 to Dimitrie Frunzetti.

p. 561 (153) *U. S. Pat.* 1733494 of Oct. 29, 1929 to Lester Kirschbraun; *French Pat.* 705505 of Nov. 5, 1930 to S. W. Aretz.

p. 562 (154) *U. S. Pats.* 58975 of Oct. 23, 1866 to Franklin Bearse and G. E. Hopkins; 1733495 and 1733496 of Oct. 29, 1929 to Lester Kirschbraun; *Can. Pats.* 313276 of Jul. 14, 1931 to Flintkote Co.; 402386 of Jan. 20, 1942 to Patent & Licensing Corp.; *Brit. Pats.* 312467 of May 4, 1928 to F. W. Gough; 341443 of Oct. 9, 1929 to N. V. de Bataafsche Petroleum Maatschappij.

p. 562 (155) *U. S. Pats.* 1408224 of Feb. 28, 1922 to C. S. Reeve; 2137226 of Nov. 22, 1938 to U. B. Bray and L. B. Beckwith; *Brit. Pats.* 486932 of Jun. 13, 1938 to Standard Oil Development Co.; 494380 of Jan. 5, 1938 to Dimitrie Frunzetti; 499352 of Apr. 26, 1938 to Soc. anom. de la Route; *Ger. Pat.* 601891 of Aug. 18, 1931 to Paul Lechler; *French Pat.* 529956 of Feb. 6, 1925 to F. Simon; *Australian Pat.* 1932/10002 of Nov. 7 to International Bitumen Emulsions Corp.

- p. 562 (156) U. S. Pats. 1469563 of Oct. 2, 1923 to Lester Kirschbraun; 1517075 of Nov. 25, 1924 to Lester Kirschbraun; 1700581 of Jan. 29, 1929 to W. E. Billingham; 1733493 of Oct. 29, 1929 to Lester Kirschbraun; 1752449 of Apr. 1, 1930 to J. S. Miller; 1793918 of Feb. 24, 1931 to J. M. Fain; 1884919 of Oct. 25, 1932 to L. G. Thompson; 1913430 of Jun. 13, 1933 to O. E. Cushman; 1948881 of Feb. 27, 1934 to Lester Kirschbraun; 2027582 of Jan. 14, 1936 to J. M. Fain; 2061076 of Nov. 17, 1936 to Lester Kirschbraun; *Can. Pats.* 279241 of Apr. 10, 1928 to Lester Kirschbraun; 310872 and 310873 of Apr. 28, 1931 to Flintkote Roads, Inc.; 315495 of Apr. 25, 1928 to G. C. Hurrell; 392345 of Nov. 5, 1940 to Patent & Licensing Corp.; *Brit. Pats.* 312580 of May 27, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 342031 of Nov. 15, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 392365 of May 18, 1933 to Paul Lechler; 400409 of Oct. 13, 1932 to F. B. Dehn; *Ger. Pat.* 569499 of May 18, 1929 to N. V. de Bataafsche Petroleum Maatschappij; *French Pats.* 684531 of Nov. 7, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 690242 of May 22, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 738596 of Jun. 14, 1932 to Paul Lechler; *Australian Pats.* 1929/23554 of Nov. 12 to N. V. de Bataafsche Petroleum Maatschappij; 1929/23761 of Nov. 21 to N. V. de Bataafsche Petroleum Maatschappij; *Hungarian Pat.* 101550 of Oct. 12, 1929 to N. V. de Bataafsche Petroleum Maatschappij; *Indian Pat.* 19028 of Jun. 27, 1932 to International Bitumen Emulsions Corp.; *Polish Pat.* 17212 of Feb. 1, 1930 to H. Hunziker.
- p. 562 (157) U. S. Pat. 1561728 of Nov. 17, 1925 to Lester Kirschbraun; *Australian Pat.* 1933/11223 of Feb. 6 to Amber Size & Chemical Co. Ltd.
- p. 562 (158) U. S. Pats. 1700581 of Jan. 29, 1929 to W. E. Billingham; 1734437 of Nov. 5, 1929 to Lester Kirschbraun; 1859517 of May 24, 1932 to Lester Kirschbraun; 2104077 of Jan. 4, 1938 to E. O. Groskopf; 2137226 of Nov. 22, 1938 to U. B. Bray and L. B. Beckwith; *Can. Pat.* 372486 of Mar. 15, 1938 to Patent & Licensing Corp.; *Brit. Pats.* 548023 of Apr. 23, 1941 to International Bitumen Emulsions, Ltd.; 549244 of May 12, 1941 to F. G. L. Becker.
- p. 562 (159) U. S. Pat. 1440356 of Dec. 26, 1922 to J. C. Morrell; *Australian Pat.* 1929/23538 of Nov. 11 to N. V. de Bataafsche Petroleum Maatschappij.
- p. 562 (160) U. S. Pat. 1733498 of Oct. 29, 1929 to Lester Kirschbraun.
- p. 562 (161) U. S. Pats. 1440356 of Dec. 26, 1922 to J. C. Morrell; 1733498 of Oct. 29, 1929 to Lester Kirschbraun; 1793957 of Feb. 24, 1931 to R. K. Painter; 1869697 of Aug. 2, 1932 to Lester Kirschbraun; 1963211 of Jan. 6, 1930 to J. M. Fain.
- p. 562 (162) *Ger. Pat. Appl.* C-160.30 of Oct. 22, 1930 to Chemieprodukte, G.m.b.H.
- p. 562 (163) *Brit. Pat.* 321344 of Nov. 17, 1928 to J. A. Montgomerie.
- p. 562 (164) U. S. Pat. 1988921 of Jan. 22, 1935 to L. E. Seng.
- p. 562 (165) U. S. Pats. 2099351, 2099352 and 2099353 of Nov. 16, 1937 to P. R. Smith.
- p. 562 (166) U. S. Pat. 163373 of May 18, 1875 to W. S. Gray and F. W. Gray; *Ger. Pats.* 583237 of Jun. 2, 1931 to Robert Tagg; *Appl. T-38952* of Jun. 1, 1931 to Robert Tagg; *French Pats.* 693448 of Apr. 7, 1930 to G. A. Lasseur and T. V. Taralon; 701730 of Sep. 5, 1930 to Robert Tagg.
- p. 562 (167) *Brit. Pat.* 344562 of Dec. 18, 1929 to J. Leben and Ormul Products, Ltd.; *French Pat.* Addition 33864 (623924) of Jul. 9, 1927 to Michel Trux.
- p. 562 (168) *French Pats.* 620755 of Dec. 8, 1927 to Asphalt Cold Mix Ltd.; Addition 36752 of Aug. 16, 1930 to Asphalt Cold Mix Ltd.; Addition 54494 of Aug. 16, 1930 to Asphalt Cold Mix Ltd.
- p. 562 (169) U. S. Pats. 1956779 of May 1, 1934 to J. W. Sparks; 2077905 of Apr. 20, 1937 to P. R. Smith; *Brit. Pats.* 292251 of Mar. 17, 1927 to Asphalt Cold Mix Ltd.; 344562 of Dec. 18, 1929 to J. Leben and Ormul Products Ltd.; *French Pats.* 594062 of Jan. 29, 1926 to F. G. Cros; 630755 of Mar. 12, 1927 to Asphalt Cold Mix, Ltd.; *Australian Pat.* 1939/108675 of Sep. 27, to H. A. Hoffman.
- p. 562 (170) U. S. Pats. 1956779 of May 1, 1934 to J. W. Sparks; 2336369 of Dec. 7, 1943 to R. P. Porter, Jr.; *Brit. Pat.* 341914 of Oct. 21, 1929 to N. V. de Bataafsche Petroleum Maatschappij.
- p. 562 (171) *Ger. Pat.* 584540 of Dec. 24, 1927 to Wilhelm Ackermann; *Swiss Pat.* 122055 of Feb. 12, 1926 to Mineral A.-G.

- p. 562 (172) U. S. Pat. 1925672 of Sep. 5, 1933 to A. A. Oeding.
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CHAPTER XXVIII

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- p. 743 (268) U. S. Pat. 1783839 of Dec. 2, 1930 to Henri Gauvin; Can. Pat. 306211 of Dec. 2, 1930 to Henri Gauvin; Ger. Pats. Designs 1393434, 1393435 and 1393436 of Apr. 18, 1936 to Emil Häfele.
- p. 743 (269) Can. Pat. 378562 of Dec. 27, 1938 to Lancaster Processes Inc.
- p. 744 (270) U. S. Pats. 86380 of Feb. 2, 1869 to H. F. Evans; 91133 of Jun. 8, 1869 to James Howard; 95689 of Oct. 12, 1869 to James Howard; 99710 of Feb. 8, 1870 to John Roberts; 146105 of Dec. 30, 1873 to J. F. Stairs; 275841 of Apr. 17, 1883 to H. M. Miner; 285490 of Sep. 25, 1883 to Josiah Jowitt; 304457 of Sep. 2, 1884 to L. L. Sagendorph; 310544 of Jan. 6, 1885 to L. L. Sagendorph; 341587 of May 11, 1886 to William Macrone; 690348 and 690349 of Dec. 31, 1901 to C. S. Bird; 758132 of Apr. 26, 1904 to G. M. Gest; 1121728 of Dec. 22, 1914 to Olav Jacobsen, Steven Troy and Halfdan Kjeldesen; 1152136 of Aug. 31, 1915 to G. R. Wyman; 1184070 of May 23, 1916 to E. J. Cady; 1381967 of Jun. 21, 1921 to C. M. Clarke; Can. Pats. 22456 of Sep. 15, 1885 to L. L. Sagendorph; 23333 of Feb. 3, 1886 to L. L. Sagendorph; Ger. Pats. 656 of Jul. 26, 1877 to C. Homberg; 54224 of Mar. 7, 1890 to L. Jacobius & Söhne; 84288 of Aug. 26, 1894 to Richard Müller; 102819 of Jul. 24, 1897 to Stephan Mattar; 103899 of Jul. 29, 1898 to Wilhelm Höpfner; 138171 of Nov. 27, 1901 to Max Alker and Walter Vinzelberg; 145586 of Oct. 18, 1902 to Hendrik de Clercq; 170063 of Apr. 1, 1905 to Schwarz'sche Maschinenfabrik und Eisengießerei Emanuel Bergmann; 177610 of Jan. 20, 1906 to Johan de Clercq; 180957 of Oct. 27, 1905 to Bergische Dachpappen- und Teerprodukten-Fabrik Gottfr. Aug. Nebeling & Comp., G.m.b.H.; 203502 of May 29, 1907 to Otto Thümmel; 231273 of Feb. 27, 1909 to Heinrich Krause; 236847 of Jan. 22, 1910 to Ernst Mallmann; 238564 of May 11, 1910 to Ernst Mallmann; 263765 of Sep. 7, 1912 to Maschinenfabrik Calbe a.S., G.m.b.H.; Design 738658 of Apr. 1, 1920 to Hermann Meyer; Design 1288687 of Jan. 4, 1934 to C. G. Haubold, A.-G.
- p. 744 (271) U. S. Pats. 1268430 of Jun. 4, 1918 to E. J. Cady; 1268446 of Jun. 4, 1918 to G. E. Ferguson; 1275216 of Aug. 13, 1918 to E. J. Cady; 1315763 of Sep. 9, 1919 to C. T. Dickey; 1331729 of Feb. 24, 1920 to J. D. Taylor; 1338624 of Apr. 27, 1920 to O. A. Heppes and C. E. Rahr; 1698886 of Jan. 15, 1929 to R. T. Johnston; 1736633 of Nov. 19, 1929 to K. H. Schutte; 1752972 of Apr. 1, 1930 to C. J. Beaver; 1842111 of Jan. 19, 1932 to C. J. Pater; 1845775 of Feb. 16, 1932 to Joseph Zavertnik, Jr. and A. A. MacCubbin; 1884486 of Oct. 25, 1932 to Joseph Zavertnik, Jr.; 1920541 of Aug. 1, 1933 to Joseph Zavertnik, Jr. and A. A. MacCubbin; 1958984 of May 15, 1934 to C. J. Beaver; 2339045 of Jan. 11, 1944 to C. J. Beaver and E. L. Davey; Can. Pats. 259398 of Mar. 30, 1926 to Flintkote Co.; 400457 and 400458 of Nov. 4, 1941 to Imperial Tobacco Co. of Canada Ltd.; 414917 of Aug. 31, 1943 to W. T. Glover & Co. Ltd.; Ger. Pats. 534059 of Mar. 7, 1929 to W. T. Glover & Co., Ltd.;

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577087 of Apr. 9, 1931 to S. D. Warren Co.; 597290 of Feb. 7, 1932 to W. T. Glover & Co., Ltd.

p. 744 (272) U. S. Pats. 1268446 of Jun. 4, 1918 to G. E. Ferguson; 1558549 of Oct. 27, 1925 to R. T. Johnston; 1587652 of Jun. 8, 1926 to R. T. Johnston; 1631826 of Jun. 7, 1927 to Lester Kirschbraun and C. E. Rahr; 1712770 of May 14, 1929 to H. C. Koch; 1751889 of Mar. 25, 1930 to C. R. Sculley; 1765777 of Jun. 24, 1930 to A. E. Schutte; Can. Pats. 244209 and 244210 of Nov. 4, 1924 to Flintkote Co. and R. T. Johnston; 295309 of Dec. 3, 1929 to C. R. Sculley; 333673 of Jul. 4, 1933 to J. J. Beaver.

p. 744 (273) U. S. Pats. 1751889 of Mar. 25, 1930 to C. R. Sculley; 1765777 of Jun. 24, 1930 to A. E. Schutte.

p. 744 (274) U. S. Pats. 1116111 of Feb. 7, 1871 to J. M. Cobb; 1435810 of Nov. 14, 1922 to J. V. Cunha; 1589537 of Jun. 22, 1927 to N. C. Pike; 1636750 of Jul. 26, 1927 to J. E. Miller; 1754024 of Apr. 8, 1930 to C. L. Keller.

p. 744 (275) "Asphalt Emulsions in Paper Making," by Joseph Rossman, *Paper Trade J.*, 89, 215 (1929); U. S. Pats. 103199 of May 17, 1870 to Samuel Kingan; 251410 of Dec. 27, 1881 to W. B. Carpenter; 253840 of Feb. 21, 1882 to W. B. Carpenter; 334974 of Jan. 26, 1886 to A. A. Oliver; 337472 of Mar. 9, 1886 to S. M. Allen; 429999 of Jun. 10, 1890 to C. A. Catlin; 436733 of Sep. 16, 1890 to J. W. Easton; 460056 of Sep. 22, 1891 to Ernst Fahrigh; 568518 of Sep. 29, 1896 to Henry Carmichael; 915860 of Mar. 23, 1909 to H. L. Hollister; 1044126 of Nov. 12, 1912 to W. D. A. Bost; 1062686 of May 27, 1913 to J. H. Amies; 1156122 of Oct. 12, 1915 to J. C. Woodley; 1177267 of Mar. 28, 1916 to R. P. Perry; 1201756 of Oct. 17, 1916 to R. P. Perry; 1204632 of Nov. 14, 1916 to J. C. Woodley and R. P. Perry; 1240524 of Sep. 18, 1917 to J. C. Woodley; 1241146 of Sep. 25, 1917 to R. P. Perry; 1288158 and 1288159 of Dec. 17, 1918 to R. P. Perry; 1293185 and 1293186 of Feb. 4, 1919 to R. P. Perry and J. M. Jack; 1296114 of Mar. 4, 1919 to R. P. Perry; 1302209 of Apr. 29, 1919 to R. P. Perry; 1305081 of May 27, 1919 to J. P. Elliott; 1305125 of May 27, 1919 to J. J. Laing and C. W. Boling; 1305404 of Jun. 3, 1919 to R. P. Perry; 1307750 of Jun. 24, 1919 to R. P. Perry; 1316591 of Sep. 23, 1919 to R. P. Perry; 1321669 of Nov. 11, 1919 to L. O. Pickett; 1338037 of Apr. 27, 1920 to R. P. Perry; 1340181 of May 18, 1920 to G. L. Oliensis and J. S. Miller, Jr.; 1344620 of Jun. 29, 1920 to F. B. Davidson; 1352796 of Sep. 14, 1920 to J. P. Elliott; 1360313 of Nov. 30, 1920 to R. P. Perry; 1360684 of Nov. 30, 1920 to L. C. Roberts; 1365711 of Jan. 18, 1921 to W. F. McKay; 1379590 of May 24, 1921 to A. L. Clapp; 1382740 of Jun. 28, 1921 to R. P. Perry; 1386276 and 1386277 of Aug. 2, 1921 to R. P. Perry; 1387219 of Aug. 9, 1921 to H. F. Weiss; 1400312 of Dec. 13, 1921 to R. P. Perry; 1406174 of Feb. 14, 1922 to J. P. Elliott; 1411330 of Apr. 4, 1922 to J. P. Elliott; Reissue 15461 of Sep. 26, 1922 to R. P. Perry; 1438966 of Dec. 19, 1922 to R. P. Perry; 1449221 of Mar. 20, 1923 to G. H. Ellis; 1467594 of Sep. 11, 1923 to H. F. Weiss; 1476570 of Dec. 4, 1923 to F. W. Adams; 1490362 of Apr. 15, 1924 to A. L. Clapp; 1498401 of Jun. 17, 1924 to R. P. Perry; 1499291 of Jun. 24, 1924 to H. M. Castner; 1511475 of Oct. 14, 1924 to K. B. Howell and C. R. Eckert; 1511949 of Oct. 14, 1924 to F. J. Commis; 1577074 of Mar. 16, 1926 to R. P. Perry and F. W. Adams; 1698298 of Jan. 8, 1929 to A. L. Clapp; 1698733 of Jan. 15, 1929 to R. P. Perry; 1708927 of Apr. 9, 1929 to Lester Kirschbraun; 1710320 of Apr. 23, 1929 to R. P. Perry; 1712002 of May 7, 1929 to J. A. Heany; 1753690 of Apr. 8, 1930 to G. A. Brown; 1769513 of Jul. 1, 1930 to A. M. Hinkson; 1771150 of Jul. 22, 1930 to E. P. Stevenson and H. A. Buron; 1778147 of Oct. 14, 1930 to E. S. Edwards; 1792098 of Feb. 10, 1931 to C. L. Keller; 1809316 of Jun. 9, 1931 to D. F. Smith, E. J. Pieper and C. C. Vogt; 1824430 of Sep. 22, 1931 to C. C. Hall; 1825869 of Oct. 6, 1931 to C. L. Keller; 1827700 and 1827701 of Oct. 13, 1931 to Lester Kirschbraun; 1844951 of Feb. 16, 1932 to H. C. Fisher, A. M. Overton and C. L. Keller; 1859414 of May 24, 1932 to E. P. Stevenson; 1875018 of Aug. 30, 1932 to M. H. Klieftho; 1877377 of Sep. 13, 1932 to Gibson Yungblut; 1878300 of Sep. 20, 1932 to E. P. Stevenson and H. A. Buron; 1900699 of Mar. 7, 1933 to G. H. Ellis; 1901382 of Mar. 14, 1933 to E. P. Stevenson; 1901930 of Mar. 21, 1933 to E. J. Pieper, D. F. Smith and C. C. Vogt; 1923888 of Aug. 22, 1933 to Thomas Robinson; 1924601 of Aug. 29, 1933 to H. C. Fisher; 1949249 of Feb. 27, 1934 to H. C. Fisher; 1953397 of Apr. 3, 1934 to E. J. Eimer; 1970426 of Aug. 14, 1934 to H. L. Levin; 1981573 of Nov. 20, 1934 to Earl Stafford; 2022311 of Nov. 26, 1935 to H. C. Fisher; 2022675 of Dec. 10, 1935 to

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H. C. Fisher; 2036466 of Apr. 7, 1936 to G. H. Ellis; 2190034 of Feb. 13, 1940 to H. L. Levin; *Can. Pats.* 245711, 245713 and 245714 of Dec. 30, 1924 to Lester Kirschbraun; 262825 of Jul. 20, 1926 to Richardson Co.; 270963 of May 24, 1927 to A. L. Clapp; 281701 of May 16, 1928 to Herbert Abraham; 291275 of Jul. 9, 1929 to World Bestos Corp.; 303662 of Sep. 2, 1930 to Richardson Co.; 319982 of Feb. 23, 1932 to Patent & Licensing Corp.; 326380 of Sep. 27, 1932 to Flintkote Corp.; 333441 and 333442 of Jun. 20, 1933 to Richardson Co.; 343186 of Jul. 17, 1934 to International Bitumen Emulsions Corp.; 366038 of May 11, 1937 to Insulite Co.; *Brit. Pats.* of 1841 (Dec. 16), 9189 to J. A. Fanshawe; of 1860 (Aug. 2), 1685 to Abraham Ripley; of 1862 (Jun. 17), 1788 to William Sinnock and John Rowley; of 1863 (Jun. 25), 1603 to William Kirrage; of 1864 (Apr. 28), 1072 to T. G. Ghislin; of 1870 (Oct. 27), 2831 to J. G. Willans; of 1876 (Mar. 27), 1309 to Caleb Taylor; of 1876 (Apr. 26), 1750 to J. Noad; of 1888 (May 30), 7933 to O. Imray; of 1893 (Mar. 28), 6565 to Siemens Cros & Co. and E. F. A. Obach; of 1897 (Apr. 23), 10181 to M. van Look; of 1898 (Mar. 30), 7678 to Ludwig Hatschek; of 1909 (Apr. 27), 9929 to J. H. Amies; of 1911 (Apr. 6), 8622 to W. H. Rymmer; of 1915 (Jun. 15), 8815 to J. C. Woodley; 102826 of Jan. 7, 1916 to J. E. Clark; 125492 of Apr. 12, 1918 to S. G. Kelsey; 157217 of Mar. 23, 1922 to Max Rogler; 165240 of Apr. 1, 1920 to Harold Wade; 166886 of Jul. 7, 1921 to Harold Wade; 167334 of Aug. 10, 1920 to G. L. Oliensis and J. S. Miller, Jr.; 174352 of Jan. 18, 1922 to Barrett Co.; 174668 of Jan. 31, 1922 to T. K. Webster; 341542 of Nov. 1, 1928 to Richardson Co.; 354001 of Jul. 13, 1929 to H. L. Becher; 383636 of May 16, 1931 to Richardson Co.; 386991 of May 16, 1931 to Richardson Co.; 388643 of Jun. 27, 1932 to F. B. Dehn; 468106 of Jun. 29, 1937 to Paper Patents Co.; 515222 of May 26, 1938 to C. Davidson & Sons Ltd.; 541180 of Apr. 13, 1940 to Papercrete Ltd. and M. G. R. Newbould; 556025 of Oct. 19, 1942 to F. Bernheim and R. E. Pickstone; *Ger. Pats.* 179577 of Jun. 10, 1904 to J. B. Granjon and J. F. J. Berchet; 253377 of Jul. 19, 1911 to Albin Baer; 296124 of Aug. 10, 1915 to W. Schmidt and E. Heuser; 337769 of Jan. 30, 1920 to Max Rogler; 376742 of Mar. 21, 1921 to F. J. Commin; 514730 of Nov. 10, 1925 to Albert Sommer; 591112 of Dec. 28, 1933 to Richardson Co.; 602292 of May 10, 1931 to Richardson Co.; 602312 of Jan. 10, 1931 to C. A. Braun; 602616 of Oct. 16, 1932 to C. A. Braun; Appl. W-46681 of Jul. 3, 1915 to J. C. Woodley (withdrawn); 613620 of Mar. 27, 1929 to International Bitumen Emulsions Corp.; 625123 of Dec. 14, 1929 to Bitumen Handelsgesellschaft m.b.H.; *French Pats.* 683976 of Oct. 26, 1929 to Richardson Co.; 714517 of Mar. 17, 1931 to Insulite Co.; *Hungarian Pat.* 100522 of Aug. 7, 1929 to Albert Sommer.

p. 744 (276) "Dispersing Asphalt in Paperboard Furnishes," by H. M. Kieckhefer, *Fiber Containers*, 28 (No. 12), 105 (1943); U. S. Pat. 1925584 of Sep. 5, 1933 to H. C. Fisher; *French Pat.* 680371 of Aug. 16, 1929 to Albert Sommer.

p. 744 (277) *Ger. Pat.* 301435 of Feb. 24, 1898 to Ludwig Hatschek.

p. 744 (278) "Used Asphalt Paper," by Fritz Hoyer, *Wochbl. Papierfabr.*, 68 (Tech. Tl.), 337 (1937).

p. 744 (279) *Can. Pats.* 410638 of Feb. 16, 1943 to Hercules Powder Co.; 413683 of Jul. 6, 1943 to Hercules Powder Co.; *Brit. Pat.* 541670 of Jul. 12, 1940 to Hercules Powder Co.

p. 744 (280) "Waterproof Paper or Board," by Lester Kirschbraun; *Paper Trade J.*, 74, 48 (1922); "Asphalt Emulsions in Paper Making," by Joseph Rossman, *Paper Trade J.*, 89, 215 (1929); "Asphalt Board," by D. P. Bailer, *Paper Mill*, 53, 10, 24 and 26 (1930); "Nach neueren Verfahren mit Asphalt emulsionen in Stoff imprägnierte Pappen, Papiere und Formkörper aus Papierstoff für elektrotechnische und ähnliche Zwecke," by Fritz Hoyer, *Asphalt u. Teer*, 31, 1034 (1931); "Die Anwendung der KB-Emulsion in der Papierindustrie," by W. H. Wobbe, *Papierfabrikant*, 29, 333 (1931); "Asphalt papiere nach neuen Herstellungsverfahren," by Fritz Hoyer, *Kunststoffe*, 22, 11 (1932); "Mit Asphalt emulsionen imprägnierte Sackpapiere," by Fritz Hoyer, *Asphalt u. Teer*, 34, 965 (1934); "Wasserdichte Wellpappen und Wellpappenpackungen," by Fritz Hoyer, *Papierfabrikant*, 32, 419 (1934); "Bitumen emulsionen in Papieren und Pappen," by H. Calmels, *La Papeterie*, 56, 58 (1934); "Use of Bitumen Emulsions in the Paper Industry," by Fritz Hoyer, *Papier-Fabr.*, 34, 275 (1936); U. S. Pats. 577135 of Feb. 16, 1897 to P. H. Holmes; 1021660 of Mar. 26, 1912 to A. L. Clapp; 1228580 of Jun. 5, 1917 to G. W. Miles; 1234315 of Jul. 24, 1917 to E. B. Eising; Reissue

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14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1401791 of Dec. 27, 1921 to Lester Kirschbraun; 1417839 of May 30, 1922 to Lester Kirschbraun; 1461445 of Jul. 10, 1923 to Lester Kirschbraun; 1479044 of Jan. 1, 1924 to Lester Kirschbraun; 1511949 of Oct. 14, 1924 to F. J. Commin; 1515821 of Nov. 18, 1924 to H. C. Avery; 1526552 of Feb. 17, 1925 to Lester Kirschbraun; 1526553 of Feb. 17, 1925 to Lester Kirschbraun and T. W. Morse; 1533100 of Apr. 14, 1925 to A. L. Clapp; 1536399 of May 5, 1925 to Lester Kirschbraun; 1537949 and 1537950 of May 19, 1925 to Lester Kirschbraun; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; 1549992 of Aug. 18, 1925 to Lester Kirschbraun; 1561728 of Nov. 17, 1925 to Lester Kirschbraun; 1563642 of Dec. 1, 1925 to T. W. Morse; 1592294 of Jul. 13, 1926 to A. L. Clapp; 1606427 and 1606428 of Nov. 9, 1926 to Lester Kirschbraun; 1615303 of Jan. 25, 1927 to Lester Kirschbraun; 1616901, 1616902 and 1616903 of Feb. 8, 1927 to Lester Kirschbraun; 1616921 of Feb. 8, 1927 to C. E. Rahr and Lester Kirschbraun; 1621791 of Mar. 22, 1927 to Lester Kirschbraun; 1657585 of Jan. 31, 1928 to R. P. Perry; 1659401 of Feb. 14, 1928 to Lester Kirschbraun; 1663095 of Mar. 20, 1928 to R. P. Perry; 1668760 of May 8, 1928 to A. L. Clapp; 1670844 of May 22, 1928 to R. R. Cone; 1672262 of Jun. 5, 1928 to Lester Kirschbraun; 1686818 of Oct. 9, 1928 to Lester Kirschbraun; 1691752 of Nov. 13, 1928 to Edmund Bache; 1700561 of Jan. 29, 1929 to F. J. Commin and A. H. J. Wright; 1707491 of Apr. 2, 1929 to R. P. Perry; 1708926 of Apr. 9, 1929 to Lester Kirschbraun; 1722431, 1722432 and 1722434 of Jul. 30, 1929 to Lester Kirschbraun; 1723355 of Aug. 6, 1929 to Lester Kirschbraun; 1723361 of Aug. 6, 1929 to H. L. Levin; 1725645, 1725646 and 1725647 of Aug. 20, 1929 to Lester Kirschbraun; 1727003 of Sep. 3, 1929 to A. L. Clapp; 1735030 of Nov. 12, 1929 to Edmund Bache; 1738509 of Dec. 3, 1929 to Lester Kirschbraun; 1747232 of Feb. 18, 1930 to Dozier Finley; 1767532 of Jun. 24, 1930 to Lester Kirschbraun; 1771744 and 1771745 of Jul. 29, 1930 to A. L. Clapp; 1777447 of Oct. 7, 1930 to Frederic Queck; 1781645 of Nov. 11, 1930 to Lester Kirschbraun; 1785357 of Dec. 16, 1930 to H. L. Levin; 1786270 of Dec. 23, 1930 to H. M. Spencer; 1825422 of Sep. 29, 1931 to R. P. Rose; 1827700 of Oct. 13, 1931 to Lester Kirschbraun; 1856946 of May 3, 1932 to W. A. Darrah; 1884761 of Oct. 25, 1932 to H. L. Levin; 1889642 and 1889643 of Nov. 29, 1932 to G. F. Davis; 1905212 of Apr. 25, 1933 to C. A. Braun; 1912511 of Jun. 6, 1933 to F. L. Carson; 1918462 of Jul. 18, 1933 to Konrad Erdmann; 1926982 of Sep. 12, 1933 to Lester Kirschbraun; 1927047 of Sep. 19, 1933 to Thomas Robinson; 1958020 of May 8, 1934 to Thomas Robinson; 2016568 of Oct. 8, 1935 to P. R. Zinser; 2026594 of Jan. 7, 1936 to H. W. Richter; 2045410 of Jun. 23, 1936 to G. A. Richter and M. O. Schur; 2057331 of Oct. 13, 1936 to H. C. Fisher and George Acus; 2168778 of Aug. 8, 1939 to J. A. Montgomerie and P. K. Archibald; *Can. Pats.* 203698 of Sep. 7, 1920 to Lester Kirschbraun; 226168 of Nov. 21, 1922 to Lester Kirschbraun; 229000 of Feb. 27, 1923 to F. J. Commin; 243952 of Oct. 28, 1924 to Lester Kirschbraun; 245539 of Dec. 23, 1924 to G. J. Manson; 245711, 245713 and 245714 of Dec. 30, 1924 to Lester Kirschbraun; 245877 of Jan. 6, 1925 to Lester Kirschbraun; 246220 of Jan. 20, 1925 to Flintkote Co.; 248144 of Mar. 31, 1925 to Lester Kirschbraun; 248219 of Mar. 31, 1925 to Lester Kirschbraun; 248540 of Apr. 7, 1925 to Flintkote Co.; 251518 of Jul. 7, 1925 to Flintkote Co.; 256080 of Dec. 8, 1925 to Lester Kirschbraun; 260604 of May 18, 1926 to Lester Kirschbraun; 264914 of Oct. 12, 1926 to Lester Kirschbraun; 276092 of Dec. 6, 1927 to Lester Kirschbraun; 281128 of Jun. 19, 1928 to Flintkote Co.; 297615 of Feb. 18, 1930 to Flintkote Co.; 305679 of Nov. 11, 1930 to Flintkote Co.; 325757 of Sep. 6, 1932 to International Paper Co.; 340620 of Apr. 3, 1934 to Standard Oil Development Co.; 340781 of Apr. 10, 1934 to International Bitumen Emulsions Corp.; 354906 of Dec. 24, 1935 to Barrett Co.; 373340 of Apr. 26, 1938 to Bennett Inc.; 409685 of Dec. 29, 1942 to Standard Oil Development Co.; *Brit. Pats.* of 1910 (May 4), 15711 to Friedrich Raschig; 151029 of Mar. 6, 1919 to Lester Kirschbraun; 162727 of May 2, 1921 to F. J. Commin; 167613 of Aug. 18, 1921 to F. J. Commin; 174114 of Sep. 7, 1920 to Lester Kirschbraun; 185816 of Jun. 6, 1921 to Lester Kirschbraun; 262828 of Dec. 14, 1926 to Hans Friedländer; 273281 of Jun. 16, 1927 to N. V. de Bataafsche Petroleum Maatschappij; 276395 of Apr. 19, 1926 to J. J. H. Sturmeijer; 359902 and 359905 of Jul. 21, 1930 to H. D. Elkington; 366521 of Oct. 30, 1930 to H. D. Elkington; 403116 of Jun. 8, 1932 to F. B. Dehn; 418253 of Sep. 13, 1933 to International Bitumen Emulsions Corp.; and A. G. Bloxam; 442045 of Jan. 23, 1936 to Cellufoam Corp.; 467422 of Dec. 16,

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1935 to International Bitumen Emulsions Ltd.; 496746 of Mar. 2, 1937 to M-B-C Emulsion Co. Aktieselskab; 509174 of Apr. 5, 1938 to Alfred Halward and Péter Murányi; **Ger. Pats.** 2041 of Nov. 16, 1877 to F. Leroy; 216753 of Sep. 6, 1906 to Julius Kathe; 296124 of Aug. 10, 1915 to Werner Schmidt and Emil Heuser; 305525 of Jul. 12, 1917 to Werner Schmidt and Emil Heuser; 321232 of Mar. 15, 1918 to Werner Schmidt and Emil Heuser; 328733 of Nov. 9, 1918 to Wäschefabrik Gebr. Simion, A.-G.; 337769 of Jan. 30, 1920 to Max Rogler; 352860 of Nov. 21, 1920 to Farbwerke vorm. Meister, Lucius & Brüning; 392901 of Mar. 27, 1921 to I. G. Farbenindustrie, A.-G.; 392902 of Aug. 6, 1921 to I. G. Farbenindustrie, A.-G.; 401546 of Jun. 2, 1921 to Lester Kirschbraun; 404070 of Nov. 2, 1917 to Eduard Dyckerhoff; 442010 of Jun. 13, 1924 to Lester Kirschbraun; 447505 of Jun. 11, 1924 to Lester Kirschbraun; 536019 of Sep. 14, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 542342 of Feb. 24, 1930 to Deutsche Heraklith, A.-G.; 542440 of Aug. 20, 1929 to Max Taudent and Hans Eggert; 569463 of Sep. 2, 1933 to Herman Doehler; Appl. B-140683 of Nov. 29, 1928 to International Bitumen Emulsions Corp.; Appl. F-130-30 of Oct. 28, 1930 to Flintkote Co.; Appl. J-42125 of Jul. 21, 1931 to I. G. Farbenindustrie, A.-G.; 614035 of Apr. 8, 1928 to International Bitumen Emulsions Corp.; 616179 of Jul. 22, 1935 to Chem. Fabrik Flörshheim, Dr. H. Noerdlinger, A.-G.; 625123 of Feb. 4, 1936 to Bitumen Handelsgesellschaft m.b.H.; 654631 of Dec. 17, 1935 to C. A. Braun; **French Pats.** 526335 of Oct. 13, 1920 to Lester Kirschbraun; 529295 of Dec. 31, 1920 to F. J. Commin; 635968 of Jun. 14, 1927 to N. V. de Bataafsche Petroleum Maatschappij; 675909 of Sep. 25, 1929 to N. V. de Bataafsche Petroleum Maatschappij; 735847 of Apr. 22, 1932 to Bitumen Investments, Inc.; 763131 of Apr. 24, 1934 to Bitumen Investments, Inc.; **Australian Pat.** 1928/17585 of Dec. 28 to J. Durward; **Austrian Pats.** 140196 of Jan. 10, 1935 to Johann Fröhlich (Firma); 153821 of Jul. 11, 1938 to Alfred Halward; **Czechoslovakian Pat.** 40856 of Jan. 2, 1929 to Eduard Prée; **Danish Pats.** 52501 of Dec. 28, 1936 to M. B. C. Emulsion Comp. A/S.; 53045 of May 3, 1937 to A. E. W. Hansen; **Indian Pat.** 20342 of Oct. 30, 1933 to International Bitumen Emulsions Corp.; **Russian Pat.** 31751 of Apr. 5, 1932 to Wsesso-jusni nantschnoissledowatelni institut bumashnoi cellulossi promischlennosti; **Swiss Pat.** 91579 of Mar. 31, 1919 to Lester Kirschbraun.

p. 744 (281) **U. S. Pats.** 103536 of May 31, 1870 to T. R. Abbot; 111611 of Feb. 7, 1871 to J. M. Cobb; 119059 of Sep. 18, 1871 to H. N. Stimson; 151683 of Jun. 9, 1874 to Elias Burnham; 230148 of Jul. 20, 1880 to G. S. Page; 825744 of Jul. 10, 1906 to A. E. Millington; 1374885 of Apr. 12, 1921 to C. S. Hathaway; **Brit. Pats.** of 1838 (Jul. 11), 7731 to John Bethell; of 1847 (Nov. 25), 11979 to William Hutchison; of 1855 (Apr. 27), 948 to R. P. Coignet; of 1869 (Jun. 29), 275 to N. C. Szerelmey; of 1873 (Apr. 25), 1499 to Peter Jensen; of 1874 (Aug. 7), 2732 to W. E. Gedge; of 1876 (Jul. 27), 3028 to G. H. Hebblethwaite; of 1880 (Oct. 9), 4107 to J. C. Mewburn; **Ger. Pats.** 656 of Jul. 26, 1877 to C. Homberg; 29444 of Jun. 17, 1884 to E. P. Louvot; 54224 of Mar. 7, 1890 to L. Jacobus & Sons; 84288 of Aug. 26, 1894 to Richard Müller; 102819 of Jul. 24, 1897 to Stephan Mattar; 131171 of Nov. 27, 1901 to Max Alker and Walter Vinzelberg; 170063 of Apr. 1, 1905 to Schwarz'sche Maschinenfabrik; 177610 of Jan. 20, 1906 to Johan de Clercq; 180958 of Oct. 27, 1906 to Bergische Dachpappen- und Teerprodukten-Fabrik; 236847 of Jan. 22, 1907 to Ernst Mallmann; 238564 of Mar. 11, 1910 to Ernst Mallmann; 263765 of Sep. 7, 1912 to Maschinenfabrik Calbe, a.S., G.m.b.H.; 284886 of Oct. 23, 1913 to Schatz & Hübner.

p. 744 (282) **U. S. Pats.** 820694 of May 15, 1906 to L. A. Bond; 876008 of Jan. 7, 1908 to F. C. Overbury; 876010 of Jan. 7, 1908 to F. C. Overbury; **Can. Pat.** 263348 of Aug. 10, 1926 to W. R. Seigle; **Brit. Pats.** of 1843 (Oct. 5), 9890 to Benedict Albino; of 1867 (Oct. 19), 2946 to J. Anderson; of 1876 (May 21), 2295 to J. S. Norrie; of 1887 (Sep. 22), 12864 to J. C. Lyman; of 1892 (Apr. 23), 7698 to A. J. Boulton; of 1893 (Jul. 28), 14551 to G. McTear; 308890 of Mar. 19, 1928 to Eikichi Sakuma; **Ger. Pats.** 122893 of May 5, 1899 to A. W. Ander-nach; 203502 of May 29, 1907 to Otto Thümmel; 276619 of Dec. 14, 1913 to Anhydatt-Lederwerke, a.G.; 295863 of Apr. 16, 1916 to Benno Schilde and Adolf Boleg; 231273 of Feb. 27, 1909 to Heinrich Krause; Design 738658 of Apr. 1, 1920 to Hermann Meyer.

p. 745 (283) "The Absorptivity of Tar-impregnating Material under Varying Conditions of Time of Manufacture," by M. J. von Mildenstein, *Teer u. Bitumen*, 40, 2 (1942).

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- p. 745 (285) U. S. Pat. 1965703 of Jul. 10, 1934 to E. H. Hyde.
- p. 747 (286) U. S. Pats. 1312682 of Aug. 12, 1919 to A. L. Clapp; 1827026 of Oct. 13, 1931 to A. G. Leonard, Jr.; 1975584 of Oct. 2, 1934 to A. G. Leonard, Jr.; Ger. Pat. 316540 of Jun. 9, 1915 to Theodor Horst and Hermann Stöcker.
- p. 747 (287) U. S. Pats. 1589537 of Jun. 22, 1926 to A. R. Lukens and N. C. Pike; 1966458 of Jun. 17, 1934 to I. J. Novak; 2044012 of Jun. 16, 1936 to I. J. Novak; 2049469 of Aug. 4, 1936 to I. J. Novak; 2104052 of Jan. 4, 1938 to I. J. Novak; 2107304 of Feb. 8, 1938 to I. J. Novak; 2246531 of Jun. 24, 1941 to I. J. Novak; Can. Pat. 345218 of Oct. 9, 1934 to Raybestos-Manhattan, Inc.
- p. 747 (288) U. S. Pats. 1381967 of Jun. 21, 1921 to C. M. Clarke; 1389630 of Sep. 6, 1921 to C. A. Cooney; 1508959 of Sep. 16, 1924 to H. A. Cumfer; 1511187 of Oct. 7, 1924 to George Ritter; 1579003 of Mar. 30, 1926 to H. C. Koch; 1588748 of Jun. 15, 1926 to H. C. Koch; 1603976 of Oct. 19, 1926 to H. M. Nicholls; 1712770 of May 14, 1929 to H. C. Koch.
- p. 747 (289) U. S. Pats. 1116111 of Feb. 7, 1871 to J. M. Cobb; 349463 of Sep. 21, 1886 to E. G. Sparks; 362547 of May 10, 1887 to Isaac Sherck and Joseph Batig, Jr.; 380915 of Apr. 10, 1888 to H. M. Miner; 1417841 of May 30, 1922 to Lester Kirschbraun; 1435810 of Nov. 14, 1922 to J. V. Cunha; 1636750 of Jul. 26, 1927 to J. E. Miller; 1754024 of Apr. 8, 1930 to C. L. Keller; 1760606 of May 27, 1930 to N. C. Pike; 1791040 of Feb. 3, 1931 to W. H. Richardson; 1816596 of Jul. 28, 1931 to Robert Maclean; 1982679 of Dec. 4, 1934 to Robert Maclean; Can. Pats. 416563, 416564, 416565 and 416566 of Nov. 23, 1943 to Certaineed Products Corp.
- p. 747 (290) U. S. Pats. 349463 of Sep. 21, 1886 to E. G. Sparks; 1277986 of Sep. 3, 1918 to W. C. Merrill.
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- p. 747 (292) U. S. Pat. 1762329 of Jun. 10, 1930 to W. G. Dudleston; Can. Pat. 356342 of Mar. 3, 1936 to T. P. Bishop.
- p. 747 (293) U. S. Pat. 2319382 of May 18, 1943 to C. E. Wilkinson.
- p. 747 (294) U. S. Pats. 1762336 of Jun. 10, 1929 to Lester Kirschbraun; 1776590 of Sep. 23, 1930 to Lester Kirschbraun; 1930646 of Oct. 17, 1933 to P. P. Gray and E. E. Werle; 2050483 of Aug. 11, 1936 to H. D. Brown; 2117355 of May 17, 1938 to J. W. Pearl; 2132863 of Oct. 11, 1938 to M. O. Schur; Can. Pats. 299376 and 299377 of Apr. 15, 1930 to Patent & Licensing Corp.; 381843 of Jun. 6, 1939 to Barrett Co.
- p. 747 (295) U. S. Pats. 1776586 of Sep. 23, 1930 to G. P. Heppes; 2023019 of Dec. 3, 1935 to G. P. Heppes; 2040514 of May 12, 1936 to J. F. Dillon; 2040529 of May 12, 1936 to J. W. Pearl; 2323487 of Jul. 6, 1943 to V. A. Rayburn; Can. Pats. 359224 and 359225 of Jul. 21, 1936 to Barrett Co.; 390131 and 390132 of Jul. 23, 1940 to Canadian Gypsum Co. Ltd.
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- p. 747 (298) U. S. Pat. 2126872 of Aug. 16, 1938 to H. A. Cumfer.
- p. 747 (299) U. S. Pats. 1286057 of Nov. 26, 1918 to R. W. E. Moore; 1831630 of Nov. 10, 1931 to F. B. Manker.
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- p. 747 (302) U. S. Pat. 230148 of Jul. 20, 1880 to G. S. Page; Ger. Pat. 112629 of May 5, 1899 to A. W. Andernach; French Pat. 343704 of Jun. 4, 1904 to J. B. Granjon and J. F. Berchet.
- p. 747 (303) U. S. Pats. 1381967 of Jun. 21, 1921 to C. M. Clarke; 2023019 of Dec. 3, 1935 to G. P. Heppes.
- p. 747 (304) U. S. Pats. 1673186 of Jun. 12, 1928 to H. A. Cumfer; 1685078 of Sep. 25, 1928 to F. W. Adams; Can. Pat. 244602 of Nov. 18, 1924 to Guyton & Cumfer Mfg. Co.

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Szerelmei; of 1871 (Apr. 19), 1035 to W. E. Newton; of 1873 (Jul. 31), 2598 to Frederic Barnett; of 1876 (May 31), 2295 to J. S. Norrie; of 1876 (Jun. 16), 2496 to J. G. Zoller and D. Scott; of 1877 (Mar. 16), 1060 to R. Taylor; of 1878 (Aug. 30), 3436 to H. H. Henson; of 1881 (Jun. 27), 2815 to A. M. Clark; of 1881 (Oct. 11), 4408 to W. O. Callender; of 1885 (Oct. 31), 13140 to J. E. A. Pierret; of 1888 (Mar. 5), 3354 to Thomas Thomson; of 1888 (Apr. 14), 5577 to W. P. Thompson; of 1889 (May 27), 8705 to A. N. Ford; of 1891 (Feb. 17), 2865 to H. F. Williams; of 1891 (Mar. 17), 4750 to Soc. anon. des Ardoisières de Deville and V. van der Heyden; of 1893 (Sep. 9), 17003 to Emille Pierret; of 1897 (Apr. 1), 8343 to J. D. Blackwell; of 1898 (Oct. 21), 22141 to Thomas Thomson; of 1899 (Apr. 6), 7237 to A. Camille; of 1899 (Jul. 22), 15125 to J. Erskine and T. M. Erskine; of 1900 (Apr. 23), 7503 to T. K. Muir; of 1902 (May 14), 11035 to W. Torkington; of 1902 (Nov. 12), 24864 to W. W. Pilkington and W. R. Ormandy; of 1907 (Mar. 27), 7373 to C. A. Peck; *Ger. Pats.* 3097 of Mar. 7, 1878 to F. A. Malchow; 14989 of Jan. 5, 1881 to Adolph Ismer; 85563 of Mar. 14, 1895 to Rudolph Wigert; 100700 of Nov. 26, 1898 to Carl Baswitz.

p. 752 (339) U. S. *Pats.* 77257 of Apr. 28, 1868 to J. H. Cole; 78014 of May 19, 1868 to Edmond Richardson and J. H. Cole; 83575 of Oct. 27, 1868 to J. J. Wiggins; 84074 of Nov. 17, 1868 to J. W. Wheeler; 91577 of Jun. 22, 1869 to H. G. Soules; 93859 of Aug. 17, 1869 to J. M. Cobb; 95974 of Oct. 19, 1869 to R. O. Benton; 97169 of Nov. 23, 1869 to B. R. Croasdale; 101473 of Apr. 5, 1870 to Samuel Kingan; 104887 of Jun. 28, 1870 to I. L. G. Rice; 113604 of Apr. 11, 1871 to J. J. Wiggins; 114139 of Apr. 25, 1871 to Dwight Hitchcock and Willis Gibbs; 114174 of Apr. 25, 1871 to D. W. McConnell and W. W. Pierce; 120153 of Oct. 24, 1871 to C. L. Fowler; 124794 of Mar. 19, 1872 to D. G. Conger; 125517 of Apr. 9, 1872 to J. H. Barker; 128367 of Jun. 25, 1872 to W. B. Davies; 151683 of Jun. 9, 1874 to Elias Burnham; 179828, 179829 and 179830 of Jul. 11, 1876 to C. M. Warren; 180081 of Jul. 18, 1876 to C. M. Warren; 180996 of Aug. 15, 1876 to C. L. Fowler; 191208 of May 22, 1877 to C. M. Warren; 282139 of Jul. 31, 1883 to Welcome White; 284891 of Sep. 11, 1883 to J. F. Perry; 345399 of Jul. 13, 1886 to C. M. Warren; 348996 of Sep. 14, 1886 to T. J. Pearce and M. W. Beardsley; 351557 of Oct. 26, 1886 to R. T. Wray and James Thomson; 358502 of Mar. 1, 1887 to G. W. Swan; 380065 of Mar. 27, 1888 to B. C. Waite; 380915 of Apr. 10, 1888 to H. M. Miner; 437033 of Sep. 23, 1890 to R. S. Merrill; 542701 of Jul. 16, 1895 to E. F. Badgley; 820694 of May 15, 1906 to L. A. Bond; 1155443 of Oct. 5, 1915 to Jean Rosen; 1273673 of Jul. 23, 1918 to Jean Rosen; *Can. Pats.* 6606 of Sep. 28, 1876 to C. M. Warren; 13457 and 13458 of Sep. 20, 1881 to C. M. Warren; 17564 of Sep. 1, 1883 to Welcome White; 97981 of Mar. 13, 1906 to H. A. Parkin; *Brit. Pats.* of 1844 (Mar. 6), 10092 to Thomas Forster; of 1863 (Jan. 27), 232 to H. H. Henson; of 1876 (Dec. 23), 4966 to O. Brooke; of 1879 (Jun. 27), 2596 to W. B. Ritchie; of 1885 (Feb. 5), 1604 to T. J. Pearce and M. W. Beardsley; of 1888 (Feb. 21), 2543 to H. G. Boston and R. Combe; of 1897 (Apr. 1), 8343 to J. D. Blackwell; *Ger. Pat.* 92308 of Apr. 12, 1895 to A. W. Andernach; *Austrian Pat.* 71551 of Apr. 25, 1916 to Schatz & Hübner.

p. 752 (340) Granulometric analyses of the talc used as "dusting finish" will show from 5 to 25 per cent of particles passing an 80-, but retained on a 100-mesh sieve, the former applying to finely ground talcs and the latter to coarsely bolted products.

p. 753 (341) "Tentative Specifications for Asphalt-Saturated Asbestos Felts for Use in Waterproofing and in Constructing Built-up Roofs" (D 250-42T), A.S.T.M. Standards 1942, II, 1299.

p. 753 (342) "Standard Specs. for Asphalt-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs" (D 226-42), A.S.T.M. Standards 1942, II, 569.

p. 753 (343) "Federal Spec. for Felt; Asphalt-Saturated (for) Flashings, Roofing, and Waterproofing" HH-F-191a, Federal Standard Stock Catalog, Section IV (Part 5), Sep. 29, 1942; E-HH-F-191a, Dec. 23, 1942.

p. 753 (344) "Standard Specs. for Coal-Tar-Saturated Roofing Felt for Use in Waterproofing and in Constructing Built-up Roofs" (D 227-42), A.S.T.M. Standards 1942, II, 572.

p. 753 (345) "Federal Spec. for Felt; Coal-Tar-Saturated (for) Roofing and Water-

p. 753 (345 contd.)

proofing" HH-F-201, Federal Standard Stock Catalog, Section IV (Part 5), Jun. 6, 1933; Amendment-1, Sep. 14, 1943; E-HH-F-201, Apr. 1, 1942.

p. 753 (346) "Standard Specs. for Woven Cotton Fabrics Saturated with Bituminous Substances for Use in Waterproofing" (D 173-42), A.S.T.M. Standards 1942, II, 574. American Assoc. State Highway Officials Standard A.A.S.H.O.: M 117-40.

p. 753 (347) "Federal Spec. for Cotton-Fabric; Woven, Asphalt-Saturated" HH-C-581a, Federal Standard Stock Catalog, Section IV (Part 5), Dec. 18, 1935.

p. 753 (348) "Federal Spec. for Cotton-Fabric; Woven, Coal-Tar-Saturated" HH-C-591, Federal Standard Stock Catalog, Section IV (Part 5), Jul. 3, 1935.

p. 754 (349) U. S. Pat. 1481430 of Jan. 22, 1924 to F. C. Overbury.

p. 756 (350) U. S. Pats. 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1536479 of May 5, 1925 to H. A. Cumfer; 1552421 of Sep. 8, 1925 to H. A. Cumfer; 1610749 of Dec. 14, 1926 to H. A. Cumfer; 1701878 of Feb. 12, 1929 to William Freegard; 1752751 of Apr. 1, 1930 to A. E. F. Moore; Can. Pats. 224111 of Sep. 26, 1922 to Guyton & Cumfer Mfg. Co.; 244601 and 244602 of Nov. 18, 1924 to Guyton & Cumfer Mfg. Co.

p. 756 (351) U. S. Pats. 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1652518 of Dec. 13, 1927 to H. A. Cumfer.

p. 756 (352) U. S. Pat. 1536479 of May 5, 1925 to H. A. Cumfer.

p. 756 (353) U. S. Pat. 1508959 of Sep. 16, 1924 to H. A. Cumfer; 1511187 of Oct. 7, 1924 to George Ritter; 1610749 of Dec. 14, 1926 to H. A. Cumfer; 2207925 of Jul. 16, 1940 to Otto Kinne.

p. 756 (354) U. S. Pats. 1459048 of Jun. 19, 1923 to H. A. Cumfer; 1830741 of Nov. 3, 1931 to David Low; 1990406 of Feb. 5, 1935 to A. O. Hurxthal and E. B. Kerst; 2043795 of Jun. 9, 1936 to C. A. Dickhaut and C. C. Willis; 2170392 of Aug. 22, 1939 to G. J. Snyder; 2295060 of Sep. 8, 1942 to L. R. Stalder; Can. Pats. 219426 of Jun. 6, 1922 to Guyton & Cumfer Mfg. Co.; 304993 of Oct. 21, 1930 to Patent & Licensing Corp.

p. 756 (355) U. S. Pats. 1400310 of Dec. 13, 1921 to H. M. Nicholls; 1525829 of Feb. 10, 1925 to Walter Rogers; 1871013 of Aug. 9, 1932 to W. M. Shakespeare; 2128190 and 2128214 of Aug. 23, 1938 to R. W. B. Reade.

p. 756 (356) U. S. Pats. 1485340 of Feb. 26, 1924 to George Ritter; 1520014 of Dec. 23, 1924 to H. A. Cumfer; 1574835 of Mar. 2, 1926 to Robert Maclean; 1995032 of Mar. 19, 1935 to A. G. Leonard, Jr.; 2112819 of Mar. 29, 1938 to G. W. Puente; 2139619 of Dec. 6, 1938 to H. C. Howell; Can. Pats. 260251 of Apr. 27, 1906 to Guyton & Cumfer Mfg. Co.; 399938 of Oct. 14, 1941 to Certainteed Products Corp.; 411769 of Apr. 13, 1943 to Certainteed Products Corp.; Ger. Pats. 207814 of Nov. 24, 1907 to Hermann Paul and Emil Ziaja; 239242 of Jun. 20, 1909 to Hermann Paul; 305489 of Jul. 18, 1917 to Maschinenfabrik Calbe a.S., G.m.b.H.; 550664 of Dec. 9, 1930 to Gose & Werner Maschinenfabrik.

p. 756 (357) U. S. Pat. 2255075 of Sep. 9, 1941 to O. V. McGrew. [See also Reference p. 541 (2).]

p. 756 (358) U. S. Pat. 1536479 of May 5, 1928 to H. A. Cumfer.

p. 758 (359) U. S. Pats. 1574889 of Mar. 2, 1926 to W. T. Hofmann; 1701878 of Feb. 12, 1929 to William Freegard; 1876761 of Sep. 13, 1932 to L. S. Rosener; 1931430 of Oct. 17, 1933 to P. W. Bur; 1931439 of Oct. 17, 1933 to A. O. Hurxthal; 2196921 of Apr. 9, 1940 to A. O. Hurxthal; Can. Pat. 391347 of Sep. 17, 1940 to American Gypsum Co. Ltd.; Ger. Pat. Design 1319360 of Oct. 17, 1934 to Otto Kinne.

p. 758 (360) U. S. Pats. 1567919 of Dec. 29, 1925 to H. A. Cumfer; 1752751 of Apr. 1, 1930 to A. E. F. Moore; 1786992 of Dec. 30, 1930 to F. G. Gronemeyer; 1862256 of Jun. 7, 1932 to H. A. Cumfer; 2327468 of Aug. 24, 1943 to W. M. Stocker; Can. Pat. 317445 of Nov. 24, 1931 to Lehon Co.; Ger. Pats. 390029 of Feb. 24, 1922 to Johann Gmeindl; Design 1366142 of Feb. 7, 1936 to P. F. Freund & Co.; Design 1376878 of May 16, 1936 to P. F. Freund & Co.

p. 758 (361) U. S. Pats. 1467841 of Sep. 11, 1923 to H. A. Cumfer; 1601784 of Oct. 5, 1926 to H. H. Wanders; 1861374 of May 31, 1932 to H. R. Wood; 1872018 of Aug. 16, 1932 to E. T. Street; 1892670 of Jan. 3, 1933 to Fred Jaeger; 2261344 of Nov. 10, 1941 to C. J. Dele-

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gard; 2350758 of Jun. 6, 1944 to H. H. A. Heuer; *Can. Pats.* 232960 of Jul. 24, 1923 to Guyton & Cumfer Mfg. Co.; 263794 of Aug. 24, 1926 to H. H. Wanders.

p. 758 (362) U. S. Pats. 1107762 of Aug. 18, 1914 to H. A. Cumfer; 1187716 of Jun. 20, 1916 to H. A. Cumfer; 1257321 of Feb. 26, 1918 to H. A. Cumfer and O. D. McFarland; 1274388 of Aug. 6, 1918 to C. A. Cooney; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1313224 of Aug. 12, 1919 to Gottfrid Hultberg; 1365741 of Jan. 18, 1921 to A. S. Speer; 1435298 of Nov. 14, 1922 to J. L. Hildebrand and H. H. Wanders; 1435353 of Nov. 14, 1922 to H. H. Wanders; 1464309 of Aug. 7, 1923 to H. A. Cumfer; 1467841 of Sep. 11, 1923 to H. A. Cumfer; 1481183 of Jan. 15, 1924 to F. A. Browne; 1492903 of May 6, 1924 to E. T. Street; 1501162 of Jul. 15, 1924 to H. A. Cumfer; 1535678 of Apr. 28, 1925 to J. E. Miller; 1548107 of Aug. 4, 1925 to E. T. Street; 1552447 of Sep. 8, 1924 to Hugo Reichel; 1567920 of Dec. 29, 1925 to H. A. Cumfer; 1569032 and 1569033 of Jan. 12, 1926 to Hugo Reichel; 1600958 of Sep. 21, 1926 to W. J. Hart and R. T. Boas; 1676911 of Jul. 10, 1928 to C. J. McDavid; 1687823 of Oct. 16, 1928 to O. D. McFarland; 1701760 of Feb. 12, 1929 to P. J. Paffen; 1744650 of Jan. 21, 1930 to E. A. Leonard; 1751562 and 1751563 of Mar. 25, 1930 to Daniel Stinger; 1780716 of Nov. 4, 1930 to E. C. Otis; 1786992 of Dec. 30, 1930 to F. G. Gronemeyer; 1841017 and 1841018 of Jan. 12, 1932 to T. H. Eickhoff; 1847767 of Mar. 1, 1932 to H. C. Koch; 1870443 of Aug. 9, 1932 to D. A. Cumfer; 1871707 of Aug. 16, 1932 to E. E. Klumpmeyer and A. A. Maxwell; 1892680 of Jan. 3, 1933 to E. S. Petersen; 1894037 of Jan. 10, 1933 to N. P. Harshberger; 1915376 of Jun. 27, 1933 to A. E. F. Moone; 1953680 of Apr. 3, 1934 to J. V. Johansen; 1986139 of Jan. 1, 1935 to D. A. Cumfer; 2008029 of Jul. 16, 1935 to O. D. McFarland; 2010887 of Aug. 13, 1935 to A. S. Peterson; Reissue 19689 of Sep. 3, 1935 to N. P. Harshberger; 2020027 of Nov. 5, 1935 to J. L. Gore; 2055738 of Sep. 29, 1936 to E. H. Venrick; 2081280 of May 25, 1937 to J. W. Pearl; 2088118 of Jul. 27, 1937 to L. C. Schillo; 2092052 of Sep. 7, 1937 to H. A. Cumfer; 2148379 of Feb. 21, 1939 to O. D. McFarland and A. O. Mickelson; 2176385 of Oct. 17, 1939 to C. O. Walper; 2258428 of Oct. 7, 1941 to L. R. Stalder; 2311627 of Feb. 23, 1943 to F. W. Adams; *Can. Pats.* 214179 of Nov. 8, 1921 to Roofing Patents Co.; 216870 of Mar. 14, 1922 to Roofing Patents Co.; 231876 of Jun. 12, 1923 to J. L. Hildebrand and H. H. Wanders; 231877 of Jun. 12, 1923 to H. H. Wanders; 312185 of Jun. 9, 1931 to Patent & Licensing Corp.; 312825 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co.; 366943 of Jun. 22, 1937 to Certainteed Products Corp.; 388700 of May 14, 1940 to Roofing Machinery Mfg. Co., Inc.; 410728 of Feb. 23, 1943 to Canadian Gypsum Co., Ltd.

p. 758, insert (363) "Federal Spec. for Roofing; Asphalt-Prepared, Smooth-Surfaced" SS-R-501, Federal Standard Stock Catalog, Section IV (Part 5), Aug. 1, 1933, E-SS-R-501, Oct. 21, 1941.

p. 758, insert (364) "Tentative Specs. for Asphalt Roofing Surfaced with Powdered Talc or Mica" (D 224-41 T), A.S.T.M. Standards 1942, II, 1311, EA-D 224, May 11, 1942.

p. 758, insert (365) "Underwriters' Laboratories Standards for Class C Sheet-Roofing and Shingles Composed of Rag-felt Saturated with Asphalt and Surfaced with Mineral-Surfacing Materials," issued by the Underwriters' Laboratories Inc., Chicago, Ill. (established and maintained by the National Board of Fire Underwriters), Revised Jan. 2, 1941.

p. 758, insert (366) "Federal Spec. for Roofing and Shingles; Asphalt-Prepared, Mineral-Surfaced" SS-R-521, Federal Standard Stock Catalog, Section IV (Part 5), Aug. 1, 1933.

p. 758, insert (367) "Tentative Specs. for Asphalt Roofing Surfaced with Coarse Mineral Granules" (D 249-42T), A.S.T.M. Standards 1942, II, 1307, EA-D 249, May 11, 1942.

p. 758, insert (368) "Tentative Specifications for Wide Salvage Asphalt Roofing Surfaced with Coarse Mineral Granules" (D 371-41T), A.S.T.M. Standards 1942, II, 1304.

p. 758, insert (369) "Federal Spec. for Roofing; Asphalt and Asbestos, Mineral-Surfaced" SS-R-511, Federal Standard Stock Catalog, Section IV (Part 5), Jul. 9, 1926.

p. 758, insert (370) "Tentative Specifications for Asphalt-Saturated and Asphalt-Coated Asbestos Felts for Use in Constructing Built-up Roofs" (D 655-42T), A.S.T.M. Standards 1942, II, 1301.

- p. 758, insert (371) "Tentative Specs. for Asphalt Shingles Surfaced with Coarse Mineral Granules" (D 225-41T), A.S.T.M. Standards 1942, II, 1314.
- p. 758, insert (372) "Tentative Specifications for Asphalt Siding Surfaced with Coarse Mineral Granules" (D 699-42T), A.S.T.M. Standards 1942, II, 1317.
- p. 758 (373) U. S. Pat. 1756947 of May 6, 1930 to R. T. Johnston; *Can. Pat.* 247138 of Feb. 24, 1925 to R. T. Johnston; *Ger. Pat.* 322986 of Aug. 8, 1916 to W. Kühmeyer-Franke & Klauer, G.m.b.H.
- p. 758 (374) U. S. Pats. 876008 and 876010 of Jan. 7, 1908 to F. C. Overbury; 1765834 of Jun. 24, 1930 to G. P. Heppes; *Brit. Pat.* of 1907 (Dec. 24), 28405 to H. W. Lake.
- p. 758 (375) U. S. Pats. 1286077 of Nov. 26, 1918 to F. C. Overbury; 1765778 of Jun. 24, 1930 to A. E. Schutte; *Can. Pat.* 188744 of Feb. 18, 1919 to F. C. Overbury (Flintkote Co.).
- p. 758 (376) U. S. Pat. 1315362 of Sep. 9, 1919 to H. A. Cumfer.
- p. 758 (377) U. S. Pat. 1392498 of Oct. 4, 1921 to O. A. Heppes and C. E. Rahr.
- p. 758 (378) U. S. Pat. 1668563 of May 8, 1928 to R. T. Johnston; *Can. Pat.* 259400 of Mar. 30, 1926 to R. T. Johnston.
- p. 758 (379) U. S. Pats. 1400310 of Dec. 13, 1921 to H. M. Nicholls; 1443971 of Feb. 6, 1923 to Alfred Anderson; 1443975 of Feb. 6, 1923 to T. F. Caldwell; 1603976 of Oct. 19, 1926 to H. M. Nicholls; 1845317 of Feb. 16, 1932 to A. E. F. Moore; *Can. Pats.* 220683 of Jul. 11, 1922 to H. M. Nicholls; 297091 of Jan. 28, 1930 to Guyton & Cumfer Mfg. Co.
- p. 759 (380) U. S. Pats. 1289507 of Dec. 31, 1918 to L. S. Mason; 1290959 of Jan. 14, 1919 to R. L. Fowler.
- p. 759 (381) U. S. Pat. 1182414 of May 9, 1916 to F. C. Overbury.
- p. 759 (382) U. S. Pat. 1892672 of Jan. 3, 1933 to W. S. Johnston.
- p. 759 (383) U. S. Pat. 1574890 of Mar. 2, 1926 to W. T. Hofmann; *Can. Pat.* 229987 of Apr. 3, 1923 to W. T. Hofmann.
- p. 759 (384) U. S. Pat. 1357920 of Nov. 2, 1920 to Herbert Abraham.
- p. 759 (385) U. S. Pats. 1131595 of Mar. 9, 1915 to C. S. Bird and G. R. Wyman; 1187259 of Jun. 13, 1916 to B. G. Casler.
- p. 759 (386) U. S. Pats. 1366146 of Jan. 18, 1921 to J. C. Woodley; 1384110 of Jul. 12, 1921 to J. C. Woodley; 1409767 and 1409768 of Mar. 14, 1922 to R. P. Perry.
- p. 759 (387) U. S. Pats. 2105531 of Jan. 18, 1938 to H. W. Greider and G. A. Fasold; 2159586 and 2159587 of May 23, 1939 to H. W. Greider and G. A. Fasold.
- p. 759 (388) U. S. Pat. 2099094 of Nov. 16, 1937 to H. C. Avery.
- p. 759 (389) U. S. Pats. 2193472 of Mar. 12, 1940 to M. C. Bothe and F. X. Pfohl; 2286145 of Jun. 9, 1942 to John Logan, Jr.
- p. 759 (390) U. S. Pat. 2171850 of Sep. 5, 1939 to H. W. Hudson; *Can. Pat.* 393172 of Dec. 17, 1940 to H. W. Hudson.
- p. 759 (391) U. S. Pat. 2118526 of May 24, 1938 to Thomas Robinson.
- p. 759 (392) U. S. Pat. 2228877 of Jan. 14, 1941 to H. C. Howell.
- p. 759 (393) *Can. Pat.* 402387 of Jan. 20, 1942 to Patent & Licensing Corp.
- p. 759 (394) *Can. Pat.* 404283 of Apr. 21, 1942 to Patent & Licensing Corp.
- p. 759 (395) U. S. Pat. 427146 of May 6, 1890 to Hermann Bormann; *Can. Pat.* 364018 of Feb. 9, 1937 to J. A. Topping.
- p. 759 (396) *Brit. Pat.* 450412 of Jan. 16, 1935 to D. L. Irwin.
- p. 759 (397) U. S. Pats. 490668 of Jan. 31, 1893 to G. S. Lee; 624976 of May 16, 1899 to R. J. Redick; 753982 of Mar. 8, 1904 to S. R. Holland; 1544144 of Jun. 30, 1925 to Dozier Finley.
- p. 759 (398) U. S. Pats. 1592760 of Jul. 13, 1926 to A. C. Fischer; 1759999 of May 27, 1930 to C. E. Rahr; 1769628 of Jul. 1, 1930 to A. C. Fischer.
- p. 759 (399) U. S. Pats. 767723 of Aug. 16, 1904 to F. W. Terpenning; 1595079 of Aug. 10, 1926 to A. C. Fischer.
- p. 759 (400) U. S. Pat. 1618192 of Feb. 22, 1927 to G. P. Heppes; *Can. Pat.* 249095 of Apr. 28, 1925 to G. P. Heppes.

- p. 760 (401) U. S. Pats. 1586892 of Jun. 1, 1926 to A. C. Fischer; 1690792 of Nov. 6, 1928 to R. C. Neptune; 1710104 of Apr. 23, 1929 to R. C. Neptune.
- p. 760 (402) Can. Pat. 221937 of Aug. 8, 1922 to J. T. Sullivan.
- p. 760 (403) U. S. Pat. 1741403 of Dec. 31, 1929 to M. L. Caton.
- p. 760 (404) U. S. Pat. 1769627 of Jul. 1, 1930 to A. C. Fischer.
- p. 760 (405) U. S. Pats. 1327933 of Jan. 13, 1920 to J. H. Young; 1410790 of Mar. 28, 1922 to J. H. Young; 1536549 of May 5, 1925 to J. H. Young; 1539512 of May 26, 1925 to Thomas Robinson; 1597168 of Aug. 24, 1926 to D. M. Sutherland, Jr.; 1640795 of Aug. 30, 1927 to J. S. Miller, Jr.; 1672579 of Jun. 5, 1928 to C. H. Rayner; 1871982 of Aug. 16, 1932 to H. D. Geyer; Can. Pats. 244618 of Nov. 18, 1924 to H. H. Robertson Co.; 282856 of Aug. 28, 1928 to Barber Asphalt Co.; Brit. Pats. 132522 of Sep. 11, 1918 to H. H. Robertson Co.; 159863 and 159864 of Jan. 4, 1921 to H. H. Robertson Co.; 209727 of Jan. 11, 1923 to J. H. Young; 243614 of Jan. 20, 1926 to C. M. Taylor.
- p. 760 (406) U. S. Pat. 314429 of Mar. 24, 1885 to W. H. H. Childs.
- p. 760 (407) U. S. Pat. 1919677 of Jul. 25, 1933 to J. H. Young.
- p. 760 (408) U. S. Pat. 1904341 of Apr. 18, 1933 to J. H. Young.
- p. 760 (409) U. S. Pats. 1800193 and 1800194 of Apr. 7, 1931 to A. C. Fischer.
- p. 760 (410) U. S. Pat. 1314753 of Sep. 2, 1919 to F. C. Overbury.
- p. 760 (411) U. S. Pat. 1238805 of Sep. 4, 1917 to F. C. Overbury.
- p. 760 (412) U. S. Pat. 2128191 of Aug. 23, 1938 to F. P. Reynolds.
- p. 760 (413) U. S. Pat. 2138456 of Nov. 29, 1938 to A. D. MacNutt.
- p. 760 (414) U. S. Pat. 1289328 of Dec. 31, 1918 to H. R. Wardell.
- p. 760 (415) U. S. Pat. 1934315 of Nov. 7, 1933 to Morris Levine; Can. Pat. 357604 of May 5, 1936 to Carborundum Co.
- p. 760 (416) U. S. Pat. 1963752 of Jun. 19, 1934 to A. D. MacNutt; Can. Pat. 365511 of Apr. 20, 1937 to Certaineed Products Corp.
- p. 760 (417) U. S. Pat. 2022429 of Nov. 26, 1935 to C. J. Merriam.
- p. 760 (418) U. S. Pat. 1808590 of Jun. 2, 1931 to A. W. Andernach; Ger. Pats. 530529 of Sep. 4, 1928 to G. A. Jahn; 531761 of Jul. 6, 1929 to G. A. Jahn; 543733 of Mar. 28, 1931 to G. A. Jahn; Design 1213324 of Mar. 15, 1932 to A. W. Andernach; Design 1228855 of Jun. 9, 1932 to A. W. Andernach; Design 1427321 of Dec. 30, 1936 to A. W. Andernach.
- p. 760 (419) Ger. Pat. 448826 of Aug. 11, 1927 to Ludwig Schwabe.
- p. 760 (420) "The Shrinkage of Roofing Paper," by A. Braeutigam, *Teer u. Bitumen*, 39, 223 (1941).
- p. 762 (421) U. S. Pat. 1447265 of Mar. 6, 1923 to A. R. Purdy; Can. Pat. 230271 of Apr. 10, 1923 to Ruberoid Co., Ltd.; Brit. Pat. 179130 of Aug. 16, 1921 to Ruberoid Co.
- p. 762 (422) U. S. Pats. 1480023, 1480024 and 1480025 of Jan. 8, 1924 to A. S. Speer; 1640678 and 1640679 of Aug. 30, 1927 to A. S. Speer; Can. Pat. 218271 of May 2, 1922 to Roofing Patents Co.
- p. 762 (423) Ger. Pats. 573115 of Aug. 11, 1928 to Alexander Malchow and Wolfgang Malchow; Design 1251409 of Oct. 29, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.; Design 1253500 of Nov. 7, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.
- p. 762 (424) Ger. Pats. 344351 of Apr. 12, 1921 to Kurt Haurwitz; Design 1234390 of Aug. 29, 1932 to Papierfabrik Krampe, A.-G.
- p. 762 (425) Ger. Pat. 600565 of Nov. 24, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.
- p. 762 (426) Ger. Pat. Design 1253499 of Nov. 7, 1932 to Reichsverband Deutscher Dachpappenfabrikanten, E. V.
- p. 762 (427) U. S. Pat. 2210348 of Aug. 6, 1940 to J. W. Swope.
- p. 762 (428) U. S. Pat. 820470 of May 15, 1906 to R. W. Bird.
- p. 763 (429) U. S. Pat. 1833471 of Nov. 24, 1931 to A. N. Parrett; Ger. Pats. 402251 of Nov. 7, 1922 to Chemisches Laboratorium für Anstrichstoffe, G.m.b.H. 414483 of May 24, 1924 to Chemisches Laboratorium für Anstrichstoffe, G.m.b.H.; 477613 of Mar. 20, 1924 to Forschungs- und Lehrinstitut für Anstrichtechnik, G.m.b.H.

p. 763 (430) U. S. Pats. 23882 of May 3, 1859 to N. A. Dyar; 43171 of Jun. 21, 1864 to S. M. Allen; 44220 of Sep. 24, 1864 to Alfred Robinson; 48311 of Jun. 20, 1865 to Alfred Robinson; Reissue 2741 of Aug. 20, 1867 to Alfred Robinson; 70028, 70029 and 70030 of Oct. 22, 1867 to John Scanlan; 75197 of Mar. 3, 1868 to Alfred Robinson; 104380 of Jun. 14, 1870 to Edward Van Orden; Reissue 4862 of Apr. 9, 1872 to Alfred Robinson; 134002 of Dec. 17, 1874 to G. W. Pond; 202902 of Apr. 23, 1878 to C. M. Warren; 209830 of Nov. 12, 1878 to Tobias New; 211669 of Jan. 28, 1879 to W. H. Rankin; 226461 of Apr. 13, 1880 to W. H. Stewart; 237158 of Feb. 1, 1881 to R. A. Bendall; 251921 of Jan. 3, 1882 to Tobias New; 256368 of Apr. 11, 1882 to G. H. Pöschel; 269786 of Dec. 26, 1882 to S. H. Hamilton; 278278 of May 22, 1883 to Augustine Sackett; 278722 of Jun. 5, 1883 to H. M. Miner; 291440 of Jan. 1, 1884 to C. M. Warren; 291600 of Jan. 8, 1884 to Josiah Jowitt; 291628 of Jan. 8, 1884 to Augustine Sackett; 293491 and 293492 of Feb. 12, 1884 to H. M. Miner; 302938 of Aug. 5, 1884 to W. H. Rankin; 312451 of Feb. 17, 1885 to Michael Ehret, Jr.; 318910 of May 26, 1885 to Josiah Jowitt; 332570 of Dec. 15, 1885 to W. H. Stewart; 341043 of May 4, 1886 to Tobias New; 351948 of Nov. 2, 1886 to C. M. Warren; 352619 of Nov. 16, 1886 to F. L. Kane; 354311 of Dec. 14, 1886 to C. A. Favel; 361050 of Apr. 12, 1887 to W. H. H. Childs; 362202 of May 3, 1887 to Philip Carey; 366857 of Jul. 19, 1887 to G. W. McGraw; 372894 of Nov. 8, 1887 to W. H. H. Childs; 373085 of Nov. 15, 1887 to H. M. Miner; Reissue 11017 of Jul. 30, 1889 to J. A. McGraw, H. L. McGraw, G. W. McGraw, Jr. and C. E. McGraw; 418569 of Dec. 31, 1889 to H. W. Johns; 427124 of May 6, 1890 to M. C. Kerbaugh; 427147 of May 6, 1890 to Hermann Bormann; 429885 of Jun. 10, 1890 to W. H. H. Childs; 453979 of Jun. 9, 1891 to G. S. Lee; 455000 of Jun. 30, 1891 to M. C. Kerbaugh; 565336 of Aug. 4, 1896 to Michael Ehret, Jr.; 800320 of Sep. 26, 1905 to T. F. Odell; 851331 of Apr. 23, 1907 to H. R. Wardell; 1083243 of Dec. 30, 1913 to W. C. Edwards, Jr.; 1154875 of Sep. 28, 1915 to James Meade; 1320549 of Nov. 4, 1919 to O. A. Heppes; 1483711 of Feb. 12, 1924 to Maurice Blumenthal; 1606428 of Nov. 9, 1926 to Lester Kirschbraun; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1644652 of Oct. 4, 1927 to Lester Kirschbraun; 1676351 of Jul. 10, 1928 to Thomas Robinson; 1694523 of Dec. 11, 1928 to J. F. White; 1788121 of Jan. 6, 1931 to F. C. Overbury; 1889177 of Nov. 29, 1932 to C. L. Keller; 2212122 of Aug. 20, 1940 to S. P. Miller; *Can. Pats.* 16821 of May 5, 1883 to G. H. Pöschel; 22642 of Oct. 19, 1885 to D. G. Conger; 38156 of Jan. 26, 1892 to H. W. Johns; 64448 of Oct. 18, 1899 to J. W. Paterson; 106875 of Aug. 6, 1907 to Barber Asphalt Paving Co.; *Brit. Pats.* of 1860 (Oct. 31), 2666 to J. Anderson; of 1868 (Apr. 22), 1308 to T. Whittaker; of 1873 (Sep. 26), 3147 to J. A. Turner; of 1874 (Feb. 4), 447 and 449 to John Macintosh; of 1874 (Mar. 4), 770 to F. Wirth; of 1880 (Oct. 9), 4107 to J. C. Mewburn; of 1882 (Jul. 25), 3539 to Josiah Jowitt and G. S. Page; of 1893 (Jul. 28), 14551 to G. McTear; 367843 of Sep. 23, 1930 to F. B. Dehn; 510031 of Jul. 26, 1939 to Binny & Co., Ltd.; *Ger. Pats.* 24612 of Oct. 5, 1882 to David Röhm; 121436 of May 6, 1899 to A. W. Andernach; *Appl. W-51.30* of Apr. 30, 1930 to Fabrik Walter-Falckenberg Nachf. (rejected); *Austrian Pat.* 4000 of Apr. 25, 1901 to Wenzel Němeček; *Swiss Pat.* 17864 of Feb. 8, 1899 to J. Gredig.

p. 763 (431) U. S. Pats. 81641 of Sep. 1, 1868 to H. W. Johns; 906252 of Dec. 8, 1908 to J. E. Meek; 1296324 of Mar. 4, 1919 to J. A. Scharwath; 1436914 of Nov. 28, 1922 to W. R. Seigle; *Brit. Pat.* 411672 of May 19, 1933 to D. L. Irwin.

p. 763 (432) U. S. Pats. 333738 of Dec. 29, 1885 to Francis Line; 418519 of Dec. 31, 1889 to H. W. Johns; 690526 of Jan. 7, 1902 to F. S. Miller and W. B. Davenport; 817619 of Apr. 10, 1906 to G. F. Bishopric; 917543 of Apr. 6, 1909 to A. J. Cohen; 1044558 of Nov. 19, 1912 to W. J. Moeller; 1062400 of May 20, 1913 to S. C. Irving; 1218217 of Mar. 6, 1917 to E. J. Schroder; 1220966 of Mar. 27, 1917 to O. R. Emigh; 1325546 of Dec. 23, 1919 to H. R. Wardell; 1548910 and 1548911 of Aug. 11, 1925 to J. C. Sherman; 1616921 of Feb. 8, 1927 to C. E. Rahr and Lester Kirschbraun; *Can. Pats.* 96317 of Nov. 28, 1905 to G. F. Bishopric; 146427 of Mar. 11, 1913 to W. J. Moeller; 211947 of May 24, 1921 to H. H. Robertson Co.; 278682 of Mar. 20, 1928 to Albert Roby; *Brit. Pats.* of 1889 (Dec. 31), 20976 to H. W. Johns; of 1891 (Oct. 14), 2457 to J. N. Hopper; of 1913 (May 20), 11752 to S. C. Irving; 411672 of May 19, 1933 to Ruberoid Co., Ltd.; *Ger. Pats.* 141760 of May 22, 1901 to Maurice Coutellier;

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654690 of Aug. 23, 1935 to Johann Drolshagen; French Pat. 772978 of Nov. 9, 1934 to Ruberoid Co., Ltd.

p. 763 (433) U. S. Pats. 42910 of May 24, 1864 to J. H. Green; 43171 of Jun. 21, 1864 to S. M. Allen; 70028 of Oct. 22, 1867 to John Scanlan; 118025 of Aug. 15, 1871 to William Kinsey; 125573 and 125574 of Apr. 9, 1872 to H. W. Johns; 150636 of May 5, 1874 to J. A. Turner; 278722 of Jun. 5, 1883 to H. M. Miner; 293491 of Feb. 12, 1884 to H. M. Miner; 293492 of Feb. 12, 1884 to H. M. Miner; 304744 of Sep. 9, 1884 to H. M. Miner; 322153 of Jul. 14, 1885 to L. F. Blair and J. W. Roche; 348844 of Sep. 7, 1886 to David Harger; 385057 of Jun. 26, 1888 to Alexander Jones; 624976 of May 16, 1899 to R. J. Redick; 636022 of Oct. 31, 1899 to G. D. Crabbs and W. H. Pendery; 753982 of Mar. 8, 1904 to S. R. Holland; 775968 of Nov. 29, 1904 to August Gross; 813336 of Feb. 20, 1906 to H. R. Wardell; 820470 of May 15, 1906 to R. W. Bird; 845414 of Feb. 26, 1907 to Samuel Herbert; 1040706 of Oct. 8, 1912 to L. F. Lindley; 1116697 of Dec. 1, 1914 to G. J. Hoffman; 1152798 of Sep. 7, 1915 to Julius de Long and J. B. d'Homergue; 1226904 of May 22, 1917 to R. C. Neptune; 1248909 of Dec. 4, 1917 to H. B. Pullar; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; 1642316 of Sep. 13, 1927 to H. R. Wardell; 1644050 of Oct. 4, 1927 to W. H. Cady; 2180723 of Nov. 21, 1939 to M. O. Schur and B. G. Hoos; 2181200 of Nov. 28, 1939 to L. J. Papineau; Can. Pat. 2557 of Jul. 25, 1873 to Edward Churchill; Brit. Pats. of 1859 (Aug. 24), 1936 to T. Briggs; of 1860 (Jul. 26), 1722 to W. E. Newton; of 1860 (Aug. 14), 1969 to R. D. McKibbin; of 1871 (Mar. 9), 630 to J. A. Turner; of 1871 (Nov. 4), 2971 to T. Briggs; of 1873 (Sep. 26), 3147 to J. A. Turner; of 1875 (Nov. 26), 4109 to P. McLaine; of 1888 (Apr. 14), 5577 to W. P. Thompson; of 1888 (Sep. 28), 13971 to Donald Nicoll; of 1893 (Oct. 14), 17003 to Emille Pierret; 510031 of Apr. 4, 1938 to Binny & Co., Ltd., B. G. Hoos, S. N. Haywood and H. S. Town; Ger. Pats. 97894 of Jul. 24, 1897 to J. E. Christensen (Erichsen & Menge); 119360 of Jun. 13, 1900 to Erichsen & Menge; Design 226487 of May 3, 1904 to Hoppe & Roehming; Design 339008 of Apr. 22, 1908 to Fritz Grimm; Design 377108 of Apr. 16, 1909 to Th. Fahnenstich Söhne; Design 1275323 of Aug. 9, 1933 to Emil Meyer; Swiss Pat. 35288 of Jan. 15, 1906 to Carl Hohl.

p. 763 (434) U. S. Pat. 419120 of Jan. 7, 1890 to David Harger.

p. 763 (435) U. S. Pats. 143697 of Oct. 14, 1873 to R. S. Jennings; 391237 of Oct. 16, 1888 to C. M. Garrison; 403588 of May 21, 1889 to G. A. Herdman; 407195 of Jul. 16, 1889 to C. M. Garrison; 418569 of Dec. 31, 1889 to H. W. Johns; 423042 of Mar. 11, 1890 to A. N. Ford; 446775 of Feb. 17, 1891 to J. N. Hopper; 539767 of May 21, 1895 to F. W. Coolbaugh and N. M. Goodlett; 542701 of Jul. 16, 1895 to E. F. Badgley; 767723 of Aug. 16, 1904 to F. W. Terpenning; 1074404 of Sep. 30, 1913 to W. L. Barnhart; 1201756 of Oct. 17, 1916 to R. P. Perry; 1320502 of Nov. 4, 1919 to J. T. Simpson; 1362887 of Dec. 21, 1920 to R. E. Moist; 1498351 of Jun. 17, 1924 to W. L. Clement; 1657979 of Jan. 31, 1928 to F. W. Thomas; 1844655 of Feb. 9, 1932 to Yuichi Hikasa; 2017106 of Oct. 15, 1935 to H. N. Sandell; Can. Pats. 22081 of Jul. 14, 1885 to Alfred Ford and J. A. Archer; 35535 of Nov. 29, 1890 to J. N. Hopper; 89633 of Oct. 18, 1904 to F. W. Terpenning; 140265 of May 7, 1912 to A. R. Manson; 220378 of Jul. 4, 1922 to R. E. Moist; Brit. Pats. of 1885 (Jan. 16), 626 to E. T. Bellhouse and D. Longsdon; of 1887 (Dec. 19), 17442 to Alfred Ford; of 1897 (Apr. 3), 809 to Eduard Kauert; of 1901 (Jan. 7), 400 to A. Gustorf; of 1901 (Nov. 27), 24139 to J. Scheck; of 1904 (Jul. 8), 15277 to A. Field; 116591 of Jul. 28, 1917 to J. C. Crets; 119494 of Jun. 29, 1917 to E. O. Brown; 120750 of Oct. 19, 1927 to E. O. Brown; Ger. Pats. 91809 of May 24, 1896 to Eduard Kauert; 168829 of Jan. 20, 1902 to L. Ebert; 189069 of Sep. 2, 1904 to C. F. Lau; 222959 of Mar. 10, 1909 to Alexander Wendler; 483735 of May 27, 1926 to Otto Engel; 514787 of Oct. 15, 1926 to Otto Engel; 530529 of Sep. 4, 1928 to G. A. Jahn; Appl. M-5.30 of Feb. 8, 1930 to Wilhelm Meusel; Design 56190 of Apr. 8, 1896 to Eduard Kauert; Design 86195 of Nov. 18, 1897 to Terrence Sparham and James Thompson; Design 430953 of Mar. 3, 1910 to August Hermann; Design 1273729 of Jul. 8, 1933 to Anton Funke; Design 1400264 of Feb. 3, 1937 to Johann Drolshagen; Design 1453203 of Sep. 30, 1938 to A. W. Andernach; Design 1473853 of Jul. 11, 1939 to A. W. Andernach; 717244 of Jan. 22, 1942 to Carl Haver

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and Edward Boecker; *Austrian Pats.* 202 of Sep. 25, 1899 to Josef Hasenbichler; 49797 of Sep. 11, 1911 to Hermann Schliszke.

p. 763 (436) *U. S. Pats.* 441036 of Nov. 18, 1890 to Arthur Siebel; 788358 of Apr. 25, 1905 to F. D. Jacobs; 816661 of Apr. 3, 1906 to F. D. Jacobs; Reissue 12475 of Apr. 24, 1906 to F. D. Jacobs; 836157 of Nov. 20, 1906 to P. W. Turner; 1002301 of Sep. 5, 1911 to E. T. Newsome; 1074404 of Sep. 30, 1913 to W. L. Barnhart; 1115714 of Nov. 3, 1914 to T. D. Miller; 1167949 of Jan. 11, 1916 to P. M. Stewart; 1168599 of Jan. 18, 1916 to J. H. Caffrey; Design 49171 of Jun. 13, 1916 to B. S. Annis; 1195090 of Aug. 15, 1916 to H. H. Robertson; 1236994 of Aug. 14, 1917 to P. M. Stewart; 1243808 of Oct. 23, 1917 to J. H. Caffrey; 1277755 of Sep. 3, 1918 to H. H. Robertson; 1277758 of Sep. 3, 1918 to W. W. Roney; 1288734 of Dec. 24, 1918 to P. M. Stewart; 1347852 of Jul. 27, 1920 to James Hamill; 1366999 of Feb. 1, 1921 to J. H. Young; 1418348 and 1418349 of Jun. 6, 1922 to J. H. Young; 1421338 of Jun. 27, 1922 to J. H. Young; 1432512 of Oct. 17, 1922 to J. H. Young; 1529463 of Mar. 10, 1925 to J. R. Burnside; 1611031 of Dec. 14, 1926 to Turner Henderson; 1700561 of Jan. 29, 1929 to F. J. Commis and A. H. J. Wright; 1812732 of Jun. 30, 1931 to J. H. Young; 1863186 of Jun. 14, 1932 to J. E. Burns; 1959610 of May 22, 1934 to J. H. Bowman; 1973004 of Sep. 11, 1934 to C. F. Langworthy and Helen Hughes; 1973103 of Sep. 11, 1934 to J. H. Young and D. S. Hubbell; 2068533 of Jan. 19, 1937 to A. W. Coffman; 2073334 of Mar. 9, 1937 to A. W. Coffman; 2124882 of Jul. 26, 1938 to E. F. Lundeen; 2188721 of Jan. 30, 1940 to J. F. McWhorter and H. D. Geyer; 2188722 of Jan. 30, 1940 to H. D. Geyer; 2274765 of Mar. 3, 1942 to Philip Zalkind; 2286120 of Jun. 9, 1942 to P. M. Snyder; 2308766 of Jan. 19, 1943 to H. L. Martinus; *Can. Pats.* 35797 of Jan. 16, 1891 to W. W. Green; 96171 of Nov. 21, 1905 to F. D. Jacobs; 105020 of Apr. 30, 1907 to P. W. Turner; 140827 of May 28, 1912 to H. R. Wardell; 230268 of Apr. 10, 1923 to H. H. Robertson Co.; 233564 of Aug. 14, 1923 to H. H. Robertson Co.; 236657 and 236658 of Dec. 25, 1923 to H. H. Robertson Co.; 248597 of Apr. 14, 1925 to J. R. Burnside; 354564 of Dec. 3, 1935 to Ruberoid Co., Ltd.; 402067 of Jan. 6, 1942 to H. H. Robertson Co.; 408887 of Nov. 24, 1942 to H. H. Robertson Co.; 413854 of Jul. 13, 1943 to H. H. Robertson Co.; *Brit. Pats.* of 1855 (Mar. 29), 703 to R. Johnson and W. W. Johnson; of 1891 (Oct. 14), 2457 to J. N. Hopper; of 1895 (Sep. 2), 16399 to Edward Nelson; of 1897 (Apr. 3), 809 to Eduard Kauert; of 1904 (Jun. 2), 12518 to W. L. Watson; of 1912 (Jan. 17), 1360 to H. R. Wardell; 106997 and 106998 of Jun. 3, 1916 to Asbestos Protected Metal Co.; 136984 of Mar. 24, 1919 to T. D. Miller; 156864 of Mar. 9, 1922 to H. H. Robertson Co.; 211746 of Apr. 16, 1923 to George Harrison; 214457 of Apr. 16, 1923 to George Harrison; 333978 of Jun. 6, 1929 to A. H. J. Wright; 355502 of Aug. 14, 1930 to Albert Field; 371683 of Jun. 30, 1931 to J. H. Young; 379188 of Dec. 29, 1931 to J. H. Young; 428916 of Nov. 9, 1934 to G. E. Black; 446978 of Nov. 8, 1934 to E. R. James; 492537 of Sep. 22, 1938 to Electrical Research Products Co.; 549058 and 549059 of May 2, 1941 to H. H. Robertson Co.; *Ger. Pats.* 45509 of Apr. 19, 1888 to Arthur Siebel; 508656 of Oct. 27, 1926 to Walli Sattig; Design 418900 of Nov. 16, 1909 to Friedrich Bock; Design 421178 of Feb. 8, 1910 to Michael Faist; Design 436615 of Jul. 18, 1910 to Michael Faist; Design 1330980 of Dec. 8, 1934 to Chr. H. Rang.

p. 763 (437) *U. S. Pats.* 2125363 of Aug. 2, 1938 to H. E. Voegeli; 2140691 of Dec. 20, 1938 to J. E. Crump; 2289699 of Jul. 14, 1942 to H. H. Doe; *Can. Pats.* 387859 of Apr. 9, 1940 to American Brass Co.; 387915 of Apr. 9, 1940 to Johns-Manville Corp.; *Ger. Pat.* 126208 of Nov. 17, 1899 to J. Scheck.

p. 763 (438) *U. S. Pat.* 2094898 of Oct. 5, 1937 to E. F. Lundeen; *Can. Pat.* 367216 of Jul. 6, 1937 to H. H. Robertson Co.; *Brit. Pat.* 467142 of Jun. 11, 1937 to H. H. Robertson Co.

p. 763 (439) *U. S. Pat.* 2244352 of Jun. 3, 1941 to J. H. Young and P. W. Jenkins.

p. 763 (440) *U. S. Pats.* 2235758 of Mar. 18, 1941 to E. R. Dearborn; 2336191 of Dec. 7, 1943 to R. L. Rose; *Brit. Pat.* 504672 of Apr. 28, 1939 to Paix & Cie. [See also Reference p. 770 (506).]

p. 764 (441) *U. S. Pat.* 985140 of Feb. 28, 1911 to Hedley Button.

p. 764 (442) *U. S. Pat.* 2274189 of Feb. 24, 1942 to John Congleton, Jr.

p. 764 (443) *U. S. Pats.* 1525552 of Feb. 17, 1925 to Lester Kirschbraun; 1526553 of

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Feb. 17, 1925 to Lester Kirschbraun and T. W. Morse; 1542557 of Jun. 16, 1925 to Lester Kirschbraun; *Can. Pat.* 309604 of Mar. 24, 1931 to J. B. Hamilton.

p. 764 (444) *Ger. Pat. Design* 1247194 of Dec. 17, 1932 to Ludwig Pringal.

p. 765 (445) *U. S. Pats.* 1164965 of Dec. 21, 1915 to H. S. Smalley; 1705015 of Mar. 12, 1929 to A. H. Irving; 1974314 of Sep. 18, 1934 to E. R. Schaeffer; 1984551 of Dec. 18, 1934 to E. R. Schaeffer.

p. 765 (446) *U. S. Pats.* 60381 of Dec. 11, 1866 to M. A. Johnson; 1543131 of Jun. 3, 1925 to J. A. Stough; 1642316 of Sep. 13, 1927 to H. R. Wardell; 1654846 of Jan. 3, 1928 to J. A. Stough.

p. 765 (447) *U. S. Pats.* 238991 of Mar. 15, 1881 to W. H. R. Tove; 249342 of Nov. 8, 1881 to William Hadden; 250301 of Nov. 29, 1881 to W. H. R. Tove; 342315 of May 25, 1886 to Frederick Beck; 358647 of Mar. 1, 1887 to William Campbell; 527416 of Oct. 16, 1894 to Antonio Federici; 674125 of May 14, 1901 to Phillip Semmer; 1082364 of Dec. 23, 1913 to A. S. Spiegel; 1134086 of Mar. 30, 1915 to F. C. Lowrey; 1154334 of Sep. 21, 1915 to F. C. Overbury; Design 48172 of Nov. 23, 1915 to W. P. Dun Lany; 1192601 of Jul. 25, 1916 to D. E. Boismenu and N. J. Wagner; 1194890 of Aug. 15, 1916 to A. S. Spiegel; 1214658 of Feb. 6, 1917 to W. P. Dun Lany; 1236462 of Aug. 14, 1917 to W. F. McKay; 1254481 of Jan. 22, 1918 to C. M. Clarke; 1264343 of Apr. 30, 1918 to A. S. Spiegel; 1264831 of Apr. 30, 1918 to W. F. McKay; 1265315 of May 7, 1918 to S. M. Ford; 1268105 of Jun. 4, 1918 to S. M. Ford; 1269906 of Jun. 18, 1918 to C. M. Clarke; 1290959 of Jan. 14, 1919 to R. L. Fowler; 1294252 of Feb. 11, 1919 to S. M. Ford; 1295360 of Feb. 25, 1919 to F. C. Overbury; 1300591 of Apr. 15, 1919 to Michael Ebinger; 1339327 of May 4, 1920 to S. M. Ford; 1365970 of Jan. 18, 1921 to M. H. Elvidge; 1376092 of Apr. 26, 1921 to O. A. Heppes; 1379368 of May 24, 1921 to A. S. Speer; 1384534 of Jul. 12, 1921 to L. F. Lindley and A. S. Speer; 1394149 of Oct. 18, 1921 to Harry Cumfer and O. D. McFarland; 1398272 of Nov. 29, 1921 to F. C. Overbury; 1414778 of May 2, 1922 to M. H. Elvidge; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1419169 of Jun. 13, 1922 to F. C. Overbury; 1434332 of Oct. 31, 1922 to M. H. Elvidge; 1448203 of Mar. 13, 1923 to Harry Cumfer and O. D. McFarland; 1452978 of Apr. 24, 1923 to J. S. Miller, Jr.; 1456224 of May 22, 1923 to A. E. Currier; 1484760 of Feb. 26, 1924 to H. A. Cumfer; 1513969 of Nov. 4, 1924 to Harry Cumfer and O. D. McFarland; 1517826 of Dec. 2, 1924 to H. A. Cumfer and O. D. McFarland; 1584557 of May 11, 1926 to H. C. Koch; 1609921 of Dec. 7, 1926 to S. G. Wright; 1623189 of Apr. 5, 1927 to Lester Kirschbraun; 1666429 of Apr. 17, 1928 to Charles Stolp, Jr.; 1669166 of May 8, 1928 to C. L. Keller; 1692210 of Nov. 20, 1928 to W. T. Hofmann; 1716072 of Jun. 4, 1929 to A. A. MacCubbin; 1774988 of Sep. 2, 1930 to Robert Maclean; 1791560 of Feb. 10, 1931 to G. P. Heppes; 1791571 of Feb. 10, 1931 to F. C. Overbury; 1794719 of Mar. 3, 1931 to Robert Maclean; 1820005 of Aug. 18, 1931 to Robert Maclean; 1833651 of Nov. 24, 1931 to E. R. Low; 1834917 of Dec. 1, 1931 to F. H. Gilchrist; 1834998 of Dec. 8, 1931 to M. R. Becker; 1857463 of May 10, 1932 to Robert Maclean; 1872622 of Aug. 16, 1932 to E. S. Donahue; 1872628 of Aug. 16, 1932 to J. F. Esch; 1886969 and 1886970 of Nov. 8, 1932 to A. E. F. Moone; 1898989 and 1898990 of Feb. 21, 1933 to N. P. Harshberger; 1900597 of Mar. 7, 1933 to E. C. Otis; 1900598 of Mar. 7, 1933 to E. T. Doyon; 1906471 of May 2, 1933 to B. W. Lambacher; 1915905 of Jun. 27, 1933 to A. S. Speer; 1916095 and 1916096 of Jun. 27, 1933 to D. A. Cumfer; 1927820 of Sep. 26, 1933 to C. R. Eckert; 1928274 and 1928275 of Sep. 26, 1933 to J. L. Wettlauffer; 1933878 of Nov. 7, 1933 to J. D. Stebbins; 1967419 of Jul. 24, 1934 to A. E. F. Moone; 1998078 and 1998079 of Apr. 16, 1935 to William Freegard; 2000077 of May 7, 1935 to N. P. Harshberger; 2011006 of Aug. 13, 1935 to Robert Maclean; 2037788 of Apr. 21, 1936 to Gottfried Hultberg; 2037822 of Apr. 21, 1936 to John Robert; 2068761 of Jan. 26, 1937 to B. S. Penley; 2068767 of Jan. 26, 1937 to John Robert; 2074130 of Mar. 16, 1937 to B. S. Penley; 2074131 of Mar. 16, 1937 to B. S. Penley and R. A. Holdsworth; 2074147 of Mar. 16, 1937 to R. A. Holdsworth; 2074445 of Mar. 23, 1937 to J. L. Wettlauffer; 2100830 of Nov. 30, 1937 to H. F. Altheide; 2111565 of Mar. 22, 1938 to C. T. Limerick; 2133473 of Oct. 18, 1938 to B. S. Penley; 2157944 of May 9, 1939 to A. B. Walton; 2211204 of Aug. 13, 1940 to O. C. Hall; 2348578 of May 9, 1944 to J. A. Soissa; *Can. Pats.*

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161694 of Mar. 30, 1915 to F. C. Lowrey; 210448, 210452 and 210453 of Apr. 12, 1921 to Roofing Patents Co.; 217362 of Mar. 28, 1922 to Roofing Patents Co.; 251356 of Jul. 7, 1925 to H. A. Cumfer; 255416 of Nov. 17, 1925 to H. A. Cumfer and O. D. McFarland; 260002 of Apr. 20, 1926 to Flintkote Co.; 265816 of Nov. 16, 1926 to Robert Maclean; 266193 of Nov. 30, 1926 to H. A. Cumfer and O. D. McFarland; 266656 of Dec. 14, 1926 to H. C. Koch; 270344 of May 3, 1927 to Flintkote Co.; 270843 of May 24, 1927 to Flintkote Co.; 280115 of May 15, 1928 to Robert Maclean; 286917 of Feb. 5, 1929 to D. A. Cumfer; 299769 of Apr. 29, 1930 to Patent & Licensing Corp.; 310697 of Apr. 21, 1931 to Patent & Licensing Corp.; 311966 of Jun. 2, 1931 to Patent & Licensing Corp.; 350847 of Jun. 11, 1935 to Barrett Co.; 356757, 356758, 356759 of Mar. 24, 1936 to Barrett Co.; 357588, 357589 and 357590 of May 5, 1936 to Barrett Co.; 366493 of Jun. 1, 1937 to Barrett Co.; 370366 of Dec. 7, 1937 to Barrett Co.; 372439 of Mar. 15, 1938 to Barrett Co.; 372912 of Apr. 5, 1938 to Barrett Co.; 381181 of May 2, 1939 to Patent & Licensing Corp.; 396171 of Apr. 29, 1941 to Barrett Co.; 411768 of Apr. 13, 1943 to Certaineed Products Corp.; Ger. Pats. Design 1259570 of Mar. 3, 1933 to Dessauer Dachpappen- und Teerprodukte-Fabrik, m.b.H., Mathis & Dr. Wendschuh; Design 1282963 of Nov. 8, 1933 to Alfred Elben; Design 1300519 of Dec. 12, 1933 to Fritz Werner; Design 1303247 of Nov. 11, 1933 to Fritz Werner.

p. 765 (448) U. S. Pats. 1214658 and 1214659 of Feb. 6, 1917 to W. P. Dun Lany; 1263051 of Apr. 16, 1918 to S. M. Ford; 1471043 of Oct. 16, 1923 to T. J. Lords and G. P. Lennart; 1471493 of Oct. 23, 1923 to T. J. Lords and G. P. Lennart; 1764080 of Jun. 17, 1930 to R. T. Johnston; Can. Pats. 259397 of Mar. 30, 1926 to Flintkote Co.; 266193 of Nov. 30, 1926 to H. A. Cumfer and O. D. McFarland; 356760 and 356761 of Mar. 24, 1936 to Barrett Co.; 356904 of Mar. 31, 1936 to Barrett Co.

p. 765 (449) U. S. Pats. Design 45836 of May 26, 1914 to S. H. Goldberg; 1123727 of Jan. 5, 1915 to S. H. Goldberg; 1176049 of Mar. 21, 1916 to S. H. Goldberg; 1236462 of Aug. 14, 1917 to W. F. McKay; 1250577 and 1250578 of Dec. 18, 1917 to S. H. Goldberg; 1264831 of Apr. 30, 1918 to W. F. McKay; 1298690 of Apr. 1, 1919 to S. H. Goldberg; 1345922 of Jul. 6, 1920 to S. H. Goldberg; 1365902 of Jan. 18, 1921 to S. M. Ford; 1499308 of Jun. 24, 1924 to C. E. Rahr; Can. Pats. 157859 of Sep. 15, 1914 to S. H. Goldberg; 167268 of Jan. 25, 1916 to S. H. Goldberg; Brit. Pat. of 1913 (Apr. 26), 9806 to S. H. Goldberg; Ger. Pat. 288749 of Apr. 17, 1914 to S. H. Goldberg.

p. 765 (450) U. S. Pats. 1036427 of Aug. 20, 1912 to C. S. Bird; 1080647 of Dec. 9, 1913 to W. F. McKay; 1208595 of Dec. 12, 1916 to W. F. McKay; 1219652 of Mar. 20, 1917 to W. F. McKay; 1228191 of May 29, 1917 to W. P. Dun Lany; 1294785 of Feb. 18, 1919 to S. M. Ford; 1296984 of Mar. 11, 1919 to O. D. McFarland; 1351181 of Aug. 31, 1920 to W. F. McKay; 1366146 of Jan. 18, 1921 to J. C. Woodley; 1369129 of Feb. 22, 1921 to Earl Ross; 1384110 of Jul. 12, 1921 to J. C. Woodley; 1409767 of Mar. 14, 1922 to R. P. Perry; 1440358 of Dec. 26, 1922 to I. B. Whetstone; 1454323 of May 8, 1923 to Hugh MacInnes; 1469543 of Oct. 2, 1923 to G. L. Strachan and J. I. Strachan; 1472884 of Nov. 6, 1923 to C. J. Pater; 1546782 of Jul. 21, 1925 to L. M. Ford; 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1645534 of Oct. 18, 1927 to O. W. Judkins; 1697464 of Jan. 1, 1929 to W. T. Hofmann; 1754253 of Apr. 15, 1930 to H. C. Avery; 2070571 of Feb. 16, 1937 to M. R. Beasley; 2218791 of Oct. 22, 1940 to Louis Herscovitz (Brick Roll); Can. Pats. 229986 of Apr. 3, 1923 to W. T. Hofmann; 271210 of May 31, 1927 to H. C. Avery; Brit. Pat. 237499 of Dec. 31, 1924 to F. H. Clark.

p. 765 (451) U. S. Pats. 1376092 of Apr. 26, 1921 to O. A. Heppes; 1915905 of Jun. 27, 1933 to A. S. Speer; 1952754 of Mar. 27, 1934 to N. P. Harshberger; 2018216 of Oct. 22, 1935 to R. S. Maclean; 2115172 of Apr. 26, 1938 to Lester Kirschbraun; 2205798 of Jun. 25, 1940 to L. H. Mattes; 2348223 of May 9, 1944 to M. W. Papesh; Can. Pats. 359064 of Jul. 14, 1936 to Certaineed Products Corp.; 359785 of Aug. 11, 1936 to Building Products, Ltd.

p. 765 (452) U. S. Pats. 1024549 and 1024550 of Apr. 30, 1912 to M. B. Becker; 1113116 of Oct. 6, 1914 to S. H. Goldberg; 1157664 of Oct. 26, 1915 to M. B. Becker; 1214659 of Feb. 6, 1917 to W. P. Dun Lany; 1218217 of Mar. 6, 1917 to E. J. Schroder; 1222594 of Apr. 17, 1917 to M. B. Becker; Design 51438 of Oct. 30, 1917 to A. J. Caron; 1392323 of Oct. 4, 1921

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to S. H. Goldberg; 1444550 of Feb. 6, 1923 to R. C. Neptune; 1469606 of Oct. 2, 1923 to C. E. Rahr and Lester Kirschbraun; 1502003 of Jul. 22, 1924 to Herbert Abraham; 2036329 of Apr. 7, 1936 to J. D. Giles; 2094150 of Sep. 28, 1937 to J. D. Giles; *Can. Pats.* 330053 of Feb. 7, 1933 to Patent & Licensing Corp.; 366555 and 366556 of Jun. 1, 1937 to J. D. Giles.

p. 765 (453) U. S. Pats. 1450712 of Apr. 3, 1923 to W. H. Cady; 1469606 of Oct. 2, 1923 to C. E. Rahr and Lester Kirschbraun.

p. 765 (454) U. S. Pats. 1144313 of Jun. 22, 1915 to S. G. Wright; 1157664 of Oct. 26, 1915 to M. B. Becker; 2013332 of Sep. 3, 1935 to Alfred Anderson; *Can. Pats.* 147910, 147911 and 147912 of May 13, 1913 to M. B. Becker.

p. 765 (455) U. S. Pats. 1024550 of Apr. 30, 1912 to M. B. Becker; 1174960 of Mar. 14, 1916 to M. B. Becker; 1181827 of May 2, 1916 to C. S. Bird; 1268105 of Jun. 14, 1918 to S. M. Ford; Design 52538 of Oct. 8, 1918 to T. J. Lords; 1296114 of Mar. 4, 1919 to R. P. Perry; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1472227 of Oct. 30, 1923 to F. C. Overbury; 1540944 of Jun. 9, 1925 to O. D. McFarland.

p. 765 (456) U. S. Pats. 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; 1157665 of Oct. 26, 1915 to M. B. Becker; 2056273, 2056274 and 2056275 of Oct. 6, 1936 to R. A. Holdsworth; *Can. Pat.* 330883 of Mar. 14, 1933 to Barrett Co.

p. 765 (457) U. S. Pats. 702614 of Jun. 17, 1902 to W. H. Bache; 800320 of Sep. 26, 1905 to T. F. Odell; 1182415 of May 9, 1916 to F. C. Overbury and H. C. Platts; 1203598 of Nov. 7, 1916 to S. M. Ford; 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1449745 of Mar. 27, 1923 to A. L. Clapp; 1491798 of Apr. 29, 1924 to N. P. Harshberger; 1546782 of Jul. 21, 1925 to S. M. Ford; *Can. Pats.* 179755 and 179756 of Oct. 16, 1917 to S. M. Ford; 233543 of Aug. 14, 1923 to A. L. Clapp; *Ger. Pats.* 72880 of Apr. 23, 1893 to E. Fischer; 130634 of Jun. 26, 1901 to H. N. Hansen; Design 15081 of Mar. 10, 1892 to E. Fischer.

p. 765 (458) U. S. Pats. 154843 of Sep. 8, 1874 to Rowell Colby; 449636 of Mar. 31, 1891 to J. A. Smith; 838232 of Dec. 11, 1906 to J. O. Ballentine; 1126114 of Jan. 26, 1915 to A. S. Spiegel; *Can. Pat.* 105023 of Apr. 30, 1907 to J. O. Ballentine.

p. 765 (459) U. S. Pat. 1376215 of Apr. 26, 1921 to C. C. Millard; *Austrian Pat.* 65252 of Dec. 15, 1913 to Heinrich Schwarz.

p. 765 (460) U. S. Pats. 1730065 of Oct. 1, 1929 to A. C. Fischer; 2080386 of May 11, 1937 to A. C. Fischer.

p. 767 (461) *Ger. Pat.* Design 1430621 of Mar. 24, 1937 to Heinrich Schulz.

p. 767 (462) U. S. Pats. 702614 of Jun. 17, 1902 to W. H. Bache; 742614 of Oct. 27, 1903 to J. L. M. Du Four; 875099 of Dec. 31, 1907 to F. C. Overbury; 875595 of Dec. 31, 1907 to F. C. Overbury; 876009 of Jan. 7, 1908 to F. C. Overbury; 881023 of Mar. 3, 1908 to F. C. Overbury; 881024 of Mar. 3, 1908 to F. C. Overbury; 891500 and 891501 of Jun. 23, 1908 to F. C. Overbury; 908125 of Dec. 29, 1908 to F. C. Overbury; 942660 of Dec. 7, 1909 to F. C. Overbury; 966178 of Aug. 2, 1910 to J. L. M. Du Four; 978333 and 978334 of Dec. 13, 1910 to F. C. Overbury; 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1102680 of Jul. 7, 1914 to F. C. Overbury; 1110238 of Sep. 8, 1914 to A. S. Spiegel; 1126932 of Feb. 2, 1915 to Herbert Abraham; 1130368 of Mar. 2, 1915 to R. W. Bird; 1164422 of Dec. 14, 1915 to A. S. Spiegel; 1302414 of Apr. 29, 1919 to C. C. Nerness; 1345099 of Jun. 29, 1920 to F. C. Overbury; 1433983 of Oct. 31, 1922 to Frank Christenson; 1550299 of Aug. 18, 1925 to Arthur Winding; 1583977 of May 11, 1926 to H. G. Kelly; 1596272 of Aug. 17, 1926 to G. M. Jordan; 1597135 of Aug. 24, 1926 to Lester Wittenberg; 1683016 of Sep. 4, 1928 to E. L. Bell and O. M. Beebe; 1722760 of Jul. 30, 1929 to R. C. Neptune; 2182526 of Dec. 5, 1939 to Paul Rumer; *Can. Pats.* 119710 of Jul. 27, 1909 to Flintkote Mfg. Co.; 217360 of Mar. 28, 1922 to Roofing Patents Co.; *Ger. Pats.* 42731 of Aug. 21, 1887 to C. A. Eppler (Ballo & Schoepe); 226401 of Dec. 25, 1907 to Flintkote Mfg. Co.; *Austrian Pat.* 48947 of Jul. 10, 1911 to Heinrich Schwarz.

p. 767 (463) U. S. Pats. 1778903 of Oct. 21, 1930 to R. B. Levis; 1834998 of Dec. 8, 1931 to M. R. Becker; 1860047 of May 24, 1932 to A. E. F. Moone; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1923946 of Aug. 22, 1933 to A. E. F. Moone; Reissue 18919 of Aug. 22, 1933 to R. B. Levis; 1928835 of Oct. 3, 1933 to R. B. Levis; 1959986 of May 22, 1934 to A. E. F. Moone; Reissue 19903 of Mar. 24, 1936 to N. P. Harshberger.

p. 768 (464) U. S. Pats. 1126932 of Feb. 2, 1915 to Herbert Abraham; 1314476 and 1314477 of Aug. 26, 1919 to F. C. Overbury; 1482090 of Jan. 29, 1924 to F. C. Overbury; 1599512 of Sep. 14, 1926 to W. H. Cady; 1612776 of Dec. 28, 1926 to Lester Kirschbraun; 1751945 of Mar. 25, 1930 to F. C. Overbury; 1752454 of Apr. 1, 1930 to F. C. Overbury; 1755049 of Apr. 15, 1930 to W. H. Cady; 1760873 of Jun. 3, 1930 to Lester Kirschbraun; 1765800 of Jun. 24, 1930 to F. C. Overbury; 1781877 of Nov. 18, 1930 to H. L. Levin; 1782535 and 1782536 of Nov. 25, 1930 to Lester Kirschbraun; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1802032 of Apr. 21, 1931 to F. C. Overbury; 1834954 of Dec. 8, 1931 to Lester Kirschbraun; 1841296 of Jan. 12, 1932 to F. C. Overbury; 1842448 of Jan. 26, 1932 to C. R. Eckert; 1872603 of Aug. 16, 1932 to F. C. Overbury; 1912986 of Jun. 6, 1933 to Lester Kirschbraun; 1927824 of Sep. 26, 1933 to C. R. Eckert; 1950032 of Mar. 6, 1934 to Lester Kirschbraun; 1972133 of Sep. 4, 1934 to M. S. Darrow; 1982215 of Nov. 27, 1934 to Lester Kirschbraun; 2000226 of May 7, 1935 to L. A. Fry; 2011098 of Aug. 13, 1935 to J. L. Wettlaufer; 2048597 of Jul. 21, 1936 to W. H. Cady; 2063268 of Dec. 8, 1936 to J. H. Plunkett; 2089312 of Aug. 10, 1937 to J. A. Topping; 2198095 of Apr. 23, 1940 to Benjamin Sweedler; 2293747 of Aug. 25, 1942 to H. C. Koch; Can. Pats. 203981 of Sep. 14, 1920 to Flintkote Co.; 269087 of Mar. 15, 1927 to Flintkote Co.; 292771 of Sep. 3, 1929 to Flintkote Co.; 295373 of Dec. 3, 1929 to Flintkote Co.; 311967 of Jun. 2, 1931 to Patent & Licensing Corp.; 313814 of Jul. 28, 1931 to Patent & Licensing Corp.; 315677 of Sep. 29, 1931 to Flintkote Co.; 316776 of Nov. 3, 1931 to Patent & Licensing Corp.; 330052 of Feb. 7, 1933 to Patent & Licensing Corp.; 354299 of Nov. 19, 1935 to Patent & Licensing Corp.

p. 768 (465) U. S. Pat. 1748981 of Mar. 4, 1930 to W. A. Harris.

p. 768 (466) U. S. Pat. 1805292 of May 12, 1931 to F. A. Mosher.

p. 768 (467) Can. Pat. 368953 of Sep. 28, 1937 to Certaineed Products Corp.

p. 768 (468) U. S. Pats. 1387219 of Aug. 9, 1921 to H. F. Weiss; 1589513 of Jun. 22, 1926 to A. L. Clapp; 1643373 of Sep. 27, 1927 to A. L. Clapp; 1802494 of Apr. 28, 1931 to H. C. Avery; 1902298 of Mar. 21, 1933 to H. C. Avery and Lester Kirschbraun; 1952962 of Mar. 27, 1934 to H. C. Avery; Can. Pats. 211895 of May 17, 1921 to C. F. Burgess Laboratories; 277735 of Feb. 7, 1928 to Flintkote Co.; 278172 of Feb. 28, 1928 to Flintkote Co.; 278743 of Mar. 20, 1928 to Flintkote Co.; 285689 of Dec. 18, 1928 to Beckman-Dawson Roofing Co.; 285716 of Dec. 18, 1928 to Flintkote Co.

p. 768 (469) U. S. Pats. 877019 of Jan. 21, 1908 to J. W. Troeger; 1014424 of Jan. 9, 1912 to J. W. Troeger; 1197307 of Sep. 5, 1916 to F. M. Ruschhaupt; 1273388 of Jul. 23, 1918 to J. C. Loyer and O. M. Loyer; 1294785 of Feb. 18, 1919 to S. M. Ford; 1310082 of Jul. 15, 1919 to H. G. Hose; 1352931 of Sep. 14, 1920 to Oscar Altpeter; 1412828 of Apr. 18, 1922 to B. C. Beckman and A. O. Herzog; 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1604941 of Oct. 26, 1926 to W. T. Hofmann; 1662655 of Mar. 13, 1928 to Herbert Abraham; 1716072 of Jun. 4, 1929 to A. A. MacCubbin; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1821552 of Sep. 1, 1931 to Lester Kirschbraun; 1851300 of Mar. 29, 1932 to B. C. Beckman; 1886456 of Nov. 8, 1932 to J. L. Wettlaufer; 1900940 of Mar. 14, 1933 to Lester Kirschbraun; 1984218 of Dec. 11, 1934 to Lester Kirschbraun; 2003699 of Jun. 4, 1935 to Thomas Robinson; 2150004 of Mar. 7, 1939 to C. R. MacDonald; 2193472 of Mar. 12, 1940 to M. C. Bothe and F. X. Pfohl; Can. Pats. 121520 of Oct. 26, 1909 to J. W. Troeger; 213049 of Aug. 23, 1921 to Beckman-Dawson Roofing Co.; 223166 of Aug. 29, 1922 to O. A. Altpeter; 234293 of Sep. 18, 1923 to Steven Troy; 239497 and 239498 of Apr. 22, 1924 to W. T. Hofmann; 270345 of May 3, 1927 to Flintkote Co.; 311314 of May 12, 1931 to Patent & Licensing Corp.; 319983 of Feb. 23, 1932 to Patent & Licensing Corp.; 327488 of Nov. 8, 1932 to Patent & Licensing Corp.; 341331 of May 1, 1934 to Patent & Licensing Corp.; 420670 of Jun. 6, 1944 to Johns-Manville Corp.

p. 768 (470) U. S. Pat. 2099131 of Nov. 16, 1937 to S. P. Miller.

p. 768 (471) Can. Pat. 401010 of Nov. 25, 1941 to Patent & Licensing Corp.

p. 768 (472) U. S. Pats. 1256508 of Feb. 19, 1918 to M. B. Becker; 1368947 of Feb. 15, 1921 to W. W. Lewis; 1722702 of Jul. 30, 1929 to Lester Kirschbraun and O. A. Heppes; 1756989 of May 6, 1930 to F. C. Overbury; 1760873 of Jun. 3, 1930 to Lester Kirschbraun;

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1781877 of Nov. 30, 1930 to H. L. Levin; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1801245 of Apr. 14, 1931 to E. L. Chamberlain; 1807918 of Jun. 2, 1931 to Lester Kirschbraun and O. A. Heppes; 1865579 of Jul. 5, 1932 to C. R. MacDonald; 1873209 of Aug. 23, 1932 to C. R. MacDonald; 1912986 of Jun. 6, 1933 to Lester Kirschbraun; 1913475 of Jun. 13, 1933 to R. W. Conant; 1927824 of Sep. 26, 1933 to C. R. Eckert; 1955131 of Apr. 17, 1934 to Lester Kirschbraun; 1961005 of May 29, 1934 to H. L. Levin; 1970431 of Aug. 14, 1934 to F. C. Overbury; 1989554 of Jan. 29, 1935 to Lester Kirschbraun and O. A. Heppes; 2013349 of Sep. 3, 1935 to Lester Kirschbraun; 2013351 and 2013352 of Sep. 3, 1935 to H. L. Levin; 2015929 of Oct. 1, 1935 to Ernest Goodwin; 2041419 of May 19, 1936 to Lester Kirschbraun; 2043545 of Jun. 9, 1936 to Lester Kirschbraun; 2053723 of Sep. 8, 1936 to Lester Kirschbraun; 2058578 of Oct. 27, 1936 to C. R. Eckert; 2071229 of Feb. 16, 1937 to O. A. Heppes and Lester Kirschbraun; 2074684 of Mar. 23, 1937 to C. R. Eckert; 2093803 of Sep. 21, 1937 to W. H. Cady; 2104384 of Jan. 4, 1938 to W. A. Harris; 2111761 of Mar. 22, 1938 to C. R. Eckert; 2122077 of Jun. 28, 1938 to A. L. Wall; 2137308 of Nov. 22, 1938 to C. E. Rahr; 2160787 of May 30, 1939 to R. W. B. Reade; 2171143 of Aug. 29, 1939 to C. R. Eckert; 2121341 of Aug. 20, 1940 to C. R. Eckert; 2245062 of Jun. 10, 1941 to Herbert Abraham; 2347250 of Apr. 25, 1944 to C. B. Burnett; *Can. Pats.* 283901 of Oct. 9, 1928 to Building Products Co., Ltd.; 287870 of Mar. 12, 1929 to Building Products Co., Ltd.; 287889 of Mar. 12, 1929 to Flintkote Co.; 290807 of Jun. 25, 1929 to Flintkote Co.; 295372 of Dec. 3, 1929 to Flintkote Co.; 298268 of Mar. 11, 1930 to Patent & Licensing Corp.; 302605 of Jul. 29, 1930 to Patent & Licensing Corp.; 306999 of Dec. 23, 1930 to Patent & Licensing Corp.; 311967 of Jun. 2, 1931 to Patent & Licensing Corp.; 323863 of Jul. 5, 1932 to Bird & Son, Inc.; 329789 of Jan. 31, 1933 to Barrett Co.; 330052 of Feb. 7, 1933 to Patent & Licensing Corp.; 331511 of Apr. 4, 1933 to Patent & Licensing Corp.; 341267 of May 1, 1934 to Barrett Co.; 342753 of Jul. 3, 1934 to Brantford Roofing Co., Ltd.; 346638 of Dec. 11, 1934 to Barrett Co.; 348281 of Feb. 19, 1935 to Barrett Co.; 353595 of Oct. 15, 1935 to Patent & Licensing Corp.; 359594 of Aug. 4, 1936 to Patent & Licensing Corp.; 360116 and 360117 of Aug. 25, 1936 to Patent & Licensing Corp.; 370086 of Nov. 23, 1937 to Certaineed Products Corp.; 375884 and 375885 of Aug. 16, 1938 to A. L. Wall; 381686 of May 30, 1939 to Certaineed Products Corp.; 390130 of Jul. 23, 1940 to Canadian Gypsum Co., Ltd.; 416567 of Nov. 23, 1943 to Certaineed Products Corp.

p. 768 (473) U. S. Pats. 1850680 of Mar. 22, 1932 to H. L. Levin; 1873886 of Aug. 23, 1932 to G. P. Heppes; 2009617 of Jul. 30, 1935 to N. P. Harshberger; *Can. Pats.* 311312 and 311313 of May 12, 1931 to Patent & Licensing Corp.

p. 768 (474) U. S. Pats. 2142181 of Jan. 3, 1939 to Michele Croce; 2170534 of Aug. 22, 1939 to A. D. McNutt; 2178273 of Oct. 31, 1939 to Lester Wittenberg; 2253552 of Aug. 26, 1941 to George Ritter; *Can. Pats.* 389724 and 389725 of Jul. 2, 1940 to Certaineed Products Corp.

p. 768 (475) U. S. Pats. 1365800 of Jan. 18, 1921 to E. R. Snyder; Reissue 15352 of May 9, 1922 to E. R. Snyder; 1516243 of Nov. 18, 1924 to R. P. Perry; 1742724 of Jan. 7, 1930 to R. P. Perry; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1802032 of Apr. 21, 1931 to F. C. Overbury; 1864806 of Jun. 28, 1932 to D. A. Cumfer; 1870426 of Aug. 9, 1932 to G. J. Snyder; 1873213 of Aug. 23, 1932 to F. C. Overbury; 1890017 of Dec. 6, 1932 to C. R. MacDonald; 1890018 of Dec. 6, 1932 to F. C. Overbury; 1961166 of Jun. 5, 1934 to C. E. Rahr; 1967856 of Jul. 24, 1934 to B. C. Beckman; 2122739 of Jul. 5, 1938 to W. G. Dudleston; 2198095 of Apr. 23, 1940 to Benjamin Sweedler; 2229396 of Jan. 21, 1941 to Benjamin Sweedler; *Can. Pats.* 274752 of Oct. 18, 1927 to Flintkote Co.; 287509 and 287510 of Feb. 26, 1929 to Flintkote Co.; 288077 of Mar. 19, 1929 to Flintkote Co.; 304824 of Oct. 14, 1930 to Flintkote Co.; 313814 of Jul. 28, 1931 to Patent & Licensing Corp.; 316776 of Nov. 3, 1931 to Patent & Licensing Corp.; 331511 of Apr. 4, 1933 to Patent & Licensing Corp.; 402217 of Jan. 13, 1942 to Patent & Licensing Corp.

p. 768 (476) U. S. Pat. 1673991 of Jun. 19, 1928 to F. C. Overbury; *Can. Pat.* 260003 of Apr. 20, 1926 to F. C. Overbury.

p. 768 (477) U. S. Pats. 320979 of Jun. 30, 1885 to L. B. Snow; 394033 of Dec. 4, 1888

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to S. E. Trott; 877019 of Jan. 21, 1908 to J. W. Troeger; 886912 of May 5, 1908 to C. W. Young and J. G. Burruss; 933221 of Sep. 7, 1909 to C. W. Young; 1048517 of Dec. 31, 1912 to J. G. Fox and H. W. Simms; 1053792 of Feb. 18, 1913 to J. F. Dietz; Reissue 14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1763601 of Jun. 10, 1930 to Lester Kirschbraun.

p. 768 (478) U. S. Pats. 1104998 of Jul. 28, 1914 to F. C. Overbury; 1108884 of Sep. 1, 1914 to C. S. Bird; 1153418 of Sep. 14, 1915 to C. S. Bird; 1184509 of May 23, 1916 to C. S. Bird; 1897139 of Feb. 14, 1933 to F. C. Overbury.

p. 768 (479) U. S. Pats. 1585692 and 1585693 of May 25, 1926 to Thomas Robinson; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1676351 of Jul. 10, 1928 to Thomas Robinson; 1698242 of Jan. 8, 1929 to Thomas Robinson; 1716505 and 1716506 of Jun. 11, 1929 to Thomas Robinson; 1765796 of Jun. 24, 1930 to Lester Kirschbraun; 1807435 of May 26, 1931 to Thomas Robinson; 1818009 of Aug. 11, 1931 to Thomas Robinson; 1819199 and 1819200 of Aug. 18, 1931 to Thomas Robinson; 1820834 of Aug. 25, 1931 to Thomas Robinson; 1863880 of Jun. 21, 1932 to Thomas Robinson; 1868139 of Jul. 19, 1932 to D. D. Forbes; 1873040 of Aug. 23, 1932 to Thomas Robinson; 1909318 of May 16, 1933 to Thomas Robinson; 1922501 of Aug. 15, 1933 to Thomas Robinson; 1941491 of Jan. 2, 1934 to Thomas Robinson; 1952828 of Mar. 27, 1934 to C. E. Volkhardt; 1973931 of Sep. 18, 1934 to Thomas Robinson; 2035921 of Mar. 31, 1936 to R. G. Quinn; 2057167 of Oct. 13, 1936 to J. C. Sherman; Can. Pats. 158954 of Nov. 17, 1914 to Herbert Abraham; 175623 of Mar. 13, 1917 to J. C. Loyer and O. M. Loyer; 286503 of Jan. 15, 1929 to Flintkote Co.; 301468 of Jun. 24, 1930 to Lancaster Asphalt, Inc.; 315944 of Oct. 6, 1931 to Lancaster Asphalt, Inc.; 316128 of Oct. 13, 1931 to Lancaster Asphalt, Inc.; 318879 of Jan. 12, 1932 to Lancaster Asphalt, Inc.; 322097 of May 3, 1932 to Lancaster Asphalt, Inc.; 323293 and 323294 of Jun. 14, 1932 to Lancaster Asphalt, Inc.; 388838 of May 21, 1940 to Patent & Licensing Corp.

p. 768 (480) U. S. Pat. 1638746 of Aug. 9, 1927 to Thomas Robinson.

p. 768 (481) U. S. Pat. 2161440 of Jun. 6, 1939 to E. E. Venrick.

p. 768 (482) U. S. Pats. 213644 of Apr. 12, 1938 to A. R. Bollaert; 2200341 of May 14, 1940 to C. E. Rahr.

p. 768 (483) U. S. Pat. 2139820 of Dec. 13, 1938 to G. H. Graham.

p. 768 (484) Can. Pat. 394535 of Feb. 11, 1941 to Canadian Gypsum Co., Ltd.

p. 768 (485) Can. Pats. 394536 of Feb. 11, 1941 to Canadian Gypsum Co., Ltd.; 419950 of May 2, 1944 to Canadian Gypsum Co., Ltd.

p. 769 (486) U. S. Pats. 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 1157438 of Oct. 19, 1915 to A. S. Spiegel and L. F. Lindley; 1368947 of Feb. 15, 1921 to W. W. Levis.

p. 769 (487) U. S. Pat. 1758059 of May 13, 1930 to C. E. Rahr.

p. 769 (488) U. S. Pat. 1340347 of May 18, 1920 to C. E. Rahr.

p. 769 (489) U. S. Pats. 1365800 of Jan. 18, 1921 to E. R. Snyder; Reissue 15352 of May 9, 1922 to E. R. Snyder; 1516243 of Nov. 18, 1924 to R. P. Perry; 1758059 of May 13, 1930 to C. E. Rahr; Can. Pats. 225117 of Oct. 24, 1922 to Barrett Co.; 279136 of Apr. 3, 1928 to Flintkote Co.

p. 769 (490) U. S. Pats. 1270654 of Jan. 25, 1918 to F. C. Overbury; 1452978 of Apr. 24, 1923 to J. S. Miller, Jr.; Can. Pat. 185435 of Jul. 9, 1918 to F. C. Overbury; Brit. Pat. 138879 of Sep. 3, 1914 to Flintkote Co.

p. 769 (491) U. S. Pats. 2233122 of Feb. 25, 1941 to F. B. Burns; 2302183 of Nov. 17, 1942 to F. B. Burns; Can. Pat. 396336 of May 6, 1941 to U. S. Gypsum Co., Ltd.

p. 769 (492) Can. Pat. 404507 of May 5, 1942 to Canadian Gypsum Co., Ltd.

p. 769 (493) U. S. Pat. 2228463 of Jan. 14, 1941 to Lester Kirschbraun; Can. Pat. 399983 of Oct. 14, 1941 to Patent & Licensing Corp.

p. 770 (494) See Reference p. 765 (447).

p. 770 (495) U. S. Pat. 1583563 of May 4, 1926 to Herbert Abraham; Can. Pat. 291769 of Jul. 30, 1929 to Duracolor Co.

p. 770 (496) U. S. Pats. 1440358 of Dec. 26, 1922 to I. B. Whetstone; 1445777 of Feb. 20, 1923 to T. B. Lehon; 1518988 of Dec. 9, 1924 to T. B. Lehon; 1759901 of May 27, 1930 to

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N. P. Harshberger; 2142177 of Jan. 3, 1939 to L. R. Clapp; 2190654 of Feb. 20, 1940 to F. G. Eichhorn; 2196847 of Apr. 9, 1940 to F. J. Austin; 2199660 of May 7, 1940 to F. G. Eichhorn; 2250482 of Jul. 29, 1941 to N. P. Harshberger; *Can. Pats.* 126321 of Jun. 14, 1910 to G. H. Pedlar; 238327 and 238328 of Jan. 23, 1923 to Lehon Co.

p. 770 (497) U. S. *Pats.* 1361517 of Dec. 7, 1920 to A. T. Cavey; 1429728 of Sep. 19, 1922 to C. N. Forrest; 1447290 of Mar. 6, 1923 to A. C. Fischer; 1448155 of Mar. 13, 1923 to Karl Schutte; 1494380 of May 20, 1924 to Karl Schutte; 1550310 of Aug. 18, 1925 to A. C. Fischer; 1743764 of Jan. 14, 1930 to A. C. Fischer; 1769632, 1769633 and 1769634 of Jul. 1, 1930 to A. C. Fischer; 1789284, 1789285, 1789286 and 1789287 of Jan. 13, 1931 to A. C. Fischer; 1861408 of May 31, 1932 to A. C. Fischer; 1881435 of Oct. 11, 1932 to A. C. Fischer; 1924047 of Aug. 22, 1933 to A. C. Fischer; 1939004 of Dec. 12, 1933 to A. C. Fischer; 2065439 of Dec. 22, 1936 to A. C. Fischer; 2132999 of Oct. 11, 1938 to J. A. Topping; 2210209 of Aug. 6, 1940 to Lester Kirschbraun; 2300488 of Nov. 3, 1942 to C. W. Cuno; 2317596 of Apr. 27, 1943 to A. C. Fischer; *Can. Pats.* 214157 of Nov. 8, 1921 to A. T. Cavey; 233126 of Jul. 31, 1923 to Barber Asphalt Co.; 403975 of Apr. 7, 1942 to Patent & Licensing Corp.; *Brit. Pats.* 156804 of Nov. 17, 1921 to A. T. Cavey; 353910 of Apr. 29, 1930 to Durastic Bituminous Products, Ltd.

p. 770 (498) U. S. *Pat.* 1447750 of Mar. 6, 1923 to C. S. Bird.

p. 770 (499) U. S. *Pats.* 1448155 of Mar. 13, 1923 to Karl Schutte; 1494380 of May 20, 1924 to Karl Schutte; *Can. Pat.* 249586 of May 12, 1925 to Karl Schutte.

p. 770 (500) U. S. *Pat.* 1810880 of Jun. 16, 1931 to A. C. Fischer.

p. 770 (501) U. S. *Pat.* 2206915 of Jul. 9, 1940 to S. A. Ochs.

p. 770 (502) U. S. *Pat.* 2290420 of Jul. 21, 1942 to G. A. Fasold.

p. 770 (503) "Neuere Dach-, Fuszboden- und Wandbeläge mit Bitumenverwendung," by Walter Obst, *Teer u. Bitumen*, 33, 27 (1935); U. S. *Pats.* 19627 of Mar. 16, 1858 to W. T. de Golyer; 26868 of Jan. 17, 1860 to Hiram Tucker; 136722 of Mar. 11, 1875 to S. C. Hogue; 137566 of Apr. 8, 1873 to E. R. Percy; 140945 of Jul. 15, 1873 to Charles Mueller; 191208 of May 22, 1877 to C. M. Warren; 252524 of Jan. 17, 1882 to L. L. Sagendorph; 255088 of Mar. 14, 1882 to L. L. Sagendorph; 825870 of Jul. 10, 1906 to Julius Schirra; 1138069 of May 4, 1915 to Addison Applegate; 1518337 of Dec. 9, 1924 to J. F. Makowski; 1592807 of Jul. 13, 1926 to C. N. Forrest; 1627531 of May 3, 1927 to F. A. Browne; 1700930 of Feb. 5, 1929 to C. N. Forrest; 1707255 of Apr. 2, 1929 to M. S. Darrow; 1913666 and 1913667 of Jun. 13, 1933 to N. P. Harshberger; 2044781, 2044782, 2044783, 2044784, 2044785 and 2044786 of Jun. 23, 1936 to N. P. Harshberger; 2044787 and 2044788 of Jun. 23, 1936 to N. P. Harshberger and S. A. Ochs; 2068396 of Jan. 19, 1937 to S. W. Chaffee; 2095248 and 2095249 of Oct. 12, 1937 to N. P. Harshberger; 2096242 of Oct. 19, 1937 to N. P. Harshberger; 2131044 of Sep. 27, 1938 to N. P. Harshberger and S. A. Ochs; 2133988 of Oct. 25, 1938 to N. P. Harshberger; 2156286 of May 2, 1939 to N. P. Harshberger; 2156901 of May 2, 1939 to Thomas Brady; 2185195 of Jan. 2, 1940 to N. P. Harshberger; 2191465 of Feb. 27, 1940 to N. P. Harshberger and S. A. Ochs; 2209366 of Jul. 30, 1940 to A. S. Vane; 2261638 of Nov. 4, 1941 to D. S. Beach; 2276484, 2276485, 2276486 and 2276487 of Mar. 17, 1942 to N. P. Harshberger; *Can. Pats.* 339345 and 339346 of Feb. 13, 1934 to Bakelite Building Products Co., Inc.; 363123, 363124, 363125 and 363126 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.; 372763 of Mar. 29, 1938 to Bakelite Building Products Co.; 387218 and 387219 of Mar. 5, 1940 to Bakelite Building Products Co.; 394067 and 394068 of Jan. 21, 1941 to Bakelite Building Products Co.; 403939 of Apr. 7, 1942 to Certaineed Products Co.; *Brit. Pats.* 423694 of Feb. 6, 1935 to Bakelite Building Products Co.; 461655 of Aug. 22, 1935 to Bakelite Building Products Co.; 463406 of Aug. 22, 1935 to Bakelite Building Products Co.; 465268 of May 4, 1937 to Bakelite Building Products Co.; *Ger. Pat.* 212527 of Oct. 30, 1926 to Ludwig Esselborn.

p. 770 (504) *Can. Pat.* 349310 of Apr. 2, 1935 to H. H. Robertson Co.

p. 770 (505) U. S. *Pats.* 40542 of Nov. 3, 1863 to L. S. Mills and C. H. Smith; 44821 of Oct. 25, 1864 to Joseph Rodefer; 145705 of Dec. 16, 1873 to Horace Wheeler; 175533 of Mar. 28, 1876 to C. M. Warren; 357503 of Feb. 8, 1887 to William Redett; 396292 of Jan. 15, 1899 to E. C. Richmond; 756180 of Mar. 29, 1904 to J. H. Munro; 769624 of Sep. 6, 1904

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to J. H. Munro; 769663 and 769664 of Sep. 6, 1904 to J. H. Munro; 802771 of Oct. 24, 1905 to J. H. Munro; 967542 of Aug. 16, 1910 to J. H. Munro; 1134217 of Apr. 6, 1915 to J. H. Munro; 1173678 of Feb. 29, 1916 to J. H. Munro; 1217152 of Feb. 27, 1917 to Albert Chauvin; 1229622 of Jun. 12, 1917 to V. F. Lake; 1250622 and 1250623 of Dec. 18, 1917 to J. H. Munro; 1439072 of Dec. 19, 1922 to G. R. Dean; 1439434 and 1439435 of Dec. 19, 1922 to J. H. Munro; Reissue 15820 of Apr. 15, 1924 to J. H. Munro; 1549263 of Aug. 11, 1925 to R. T. Johnston; 1558186 of Oct. 20, 1925 to V. F. Lake; 1561581 of Nov. 17, 1925 to Herbert Abraham; 1583163 of May 4, 1926 to J. H. Munro; 1588540 of Jun. 15, 1926 to A. B. Fossee; 1590385 of Jun. 29, 1926 to V. F. Lake; 1593094, 1593095 and 1593096 of Jul. 20, 1926 to J. H. Munro; 1728795 of Sep. 17, 1929 to J. H. Griffin; 1852696 of Apr. 5, 1932 to S. W. Chaffee; 1854512 of Apr. 19, 1932 to O. A. Heppes; 1936055 of Nov. 21, 1933 to Donald Heaney; 1937255 of Nov. 28, 1933 to H. P. Taylor; Reissue 19518 of Apr. 9, 1935 to S. W. Chaffee; 2149741 of Mar. 7, 1939 to W. S. Miles; 2149818 of Mar. 7, 1939 to W. B. North; 2151794 of Mar. 28, 1939 to J. B. Peebles; 2193233 of Mar. 12, 1940 to J. H. Hardy; Can. Pats. 95338 of Oct. 3, 1905 to J. H. Munro; 141514 of Jul. 2, 1912 to J. H. Munro; 244144 and 244145 of Nov. 4, 1924 to J. H. Munro; 246520 of Feb. 3, 1925 to J. H. Munro; 247684 of Mar. 17, 1925 to J. H. Munro; 248634 of Apr. 14, 1925 to J. H. Munro; 248716 of Apr. 14, 1925 to Ruberoid Co., Ltd.; 248719 and 248720 of Apr. 14, 1925 to Slate Veneer Co.; 253852 of Sep. 22, 1925 to J. H. Munro; 328693 of Dec. 20, 1932 to Patent & Licensing Corp.; Brit. Pat. 241732 of Nov. 17, 1924 to Herbert Abraham; Ger. Pat. 129850 of Jun. 12, 1901 to M. F. Sieges; Danish Pat. 19843 of Feb. 5, 1915 to A. Nielson.

p. 770 (506) U. S. Pats. 113882 of Apr. 18, 1871 to T. N. Hickcox; 153749 of Aug. 4, 1874 to Rowell Colby; 164749 of Jun. 22, 1875 to Delaplaine McDaniel; 632691 of Sep. 12, 1899 to W. C. Bates; 836157 of Nov. 20, 1906 to P. W. Turner; 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1076111 of Oct. 21, 1913 to F. H. Grant; 1094893 of Apr. 28, 1914 to F. H. Grant; 1354050 of Sep. 28, 1920 to Arthur Lovell; 1512248 of Oct. 21, 1924 to James Smith; 1513940 of Nov. 4, 1924 to James Smith; 1536550 of May 5, 1925 to J. H. Young; 1551317 and 1551318 of Aug. 25, 1925 to John Logan; 1589841 of Jun. 22, 1926 to J. A. Daly; 1593205 of Jul. 20, 1926 to J. H. Young; 1636388 of Jul. 19, 1927 to Thomas Robinson; 1638746 of Aug. 9, 1927 to Thomas Robinson; 1663565 of Mar. 27, 1928 to Thomas Robinson; 1665222 of Apr. 10, 1928 to Thomas Robinson; 1666755 of Apr. 17, 1928 to C. E. Rahr and F. C. Overbury; 1673585 of Jun. 12, 1928 to F. C. Overbury; 1698267 of Jan. 8, 1929 to Lester Kirschbraun; 1701918 of Feb. 12, 1929 to J. H. Gillis; 1701926 of Feb. 12, 1929 to Lester Kirschbraun; 1723076 of Aug. 6, 1929 to R. S. Reynolds; 1727132 of Sep. 3, 1929 to R. S. Reynolds; 1753303 of Apr. 8, 1930 to R. S. Reynolds; 1794674 of Mar. 3, 1931 to D. A. Cumfer; 1813084 of Jul. 7, 1931 to Thomas Robinson; 1813089 of Jul. 7, 1931 to W. M. Shakespeare; 1819270 of Aug. 8, 1931 to W. M. Shakespeare; 1822410 of Sep. 8, 1931 to H. C. Macan; 1826761 of Oct. 13, 1931 to D. F. Fradette; 1829886 of Nov. 3, 1931 to C. E. Yates and H. C. Macan; 1864055 of Jun. 21, 1932 to W. H. Finkeldey; 1865959 of Jul. 5, 1932 to T. C. Prouty; 1871067 and 1871068 of Aug. 9, 1932 to H. C. Macan; 1908127 of May 9, 1933 to W. T. Deacon; 1909862 of May 16, 1933 to I. C. Honegger; 1927586 of Sep. 19, 1933 to D. F. Fradette; 2016429 of Oct. 8, 1935 to H. P. Hayden; 2032275 of Feb. 25, 1936 to D. F. Fradette; 2043271 of Jun. 9, 1936 to J. H. Wahl and P. H. Doe; 2050248 of Aug. 11, 1936 to J. B. Eisen; 2086794 of Jul. 13, 1937 to D. F. Fradette; 2106624 of Jan. 25, 1938 to G. I. Ray; 2114362 of Apr. 19, 1938 to A. H. Tashjian; 2134034 of Oct. 25, 1938 to H. H. Doe; 2184328 of Dec. 26, 1939 to H. F. Wildman; 2192810 of Mar. 5, 1940 to E. H. Angier; 2192814 of Mar. 5, 1940 to Samuel Gessler; 2219450 of Oct. 29, 1940 to E. H. Koenig; 2227205 of Dec. 31, 1940 to J. D. Tennison; 2249698 of Jul. 15, 1941 to P. A. Wilkins; 2289699 of Jul. 14, 1942 to H. H. Doe; Can. Pats. 152970 of Jan. 6, 1914 to F. H. Grant; 227922 of Jan. 16, 1923 to E. A. Bradshaw; 236658 of Dec. 25, 1923 to H. H. Robertson Co.; 249326 of May 5, 1925 to Anaconda Sales Co.; 249558 of May 12, 1925 to Anaconda Sales Co.; 253276 of Sep. 1, 1925 to J. H. Gillis; 255122 of Nov. 3, 1925 to Anaconda Sales Co.; 255725 and 255760 of Nov. 24, 1925 to Anaconda Sales Co.; 261530 of Jun. 8, 1926 to Anaconda Sales Co.; 285451 of Dec. 11, 1928 to I. N. Lewis; 309214 of Mar. 10, 1931 to Anaconda Sales Co.; 311037 and 311038 of May 5, 1931 to Anaconda Sales Co.;

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324387 of Jul. 19, 1932 to Singmaster & Breyer, Inc.; 327879 of Nov. 22, 1932 to H. H. Robertson Co.; 364986 of Mar. 23, 1937 to D. F. Fradette; 385828 of Dec. 26, 1939 to A. W. Pattiani (metal or wood); 387472 of Mar. 19, 1940 to Clements Batcheller; Brit. Pats. 215953 of May 8, 1923 to H. H. Robertson Co.; 220644 of Aug. 15, 1924 to International Copperclad Co.; 220646 of Aug. 15, 1924 to J. H. Gillis; 220647 of Aug. 15, 1924 to International Copperclad Co.; 307144 of Dec. 24, 1927 to J. A. Montgomerie; 340168 of Feb. 17, 1930 to International Copperclad Co.; 415416 of Feb. 27, 1933 to Hermann von Forster; 415739 of Feb. 27, 1933 to Hermann von Forster; 446943 of May 8, 1936 to N. V. Internationale Alfol Maatschappij; Ger. Pats. 249986 of Aug. 30, 1910 to Naumann Scheffel; 566110 of Feb. 26, 1930 to International Copperclad Co.; Appl. M-110358 of May 25, 1929 to Vereinigte Deutsche Metallwerke, A.-G.; Appl. M-112940 of Nov. 30, 1929 to Vereinigte Deutsche Metallwerke, A.-G.; Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.; Design 1213311 of Mar. 2, 1932 to Carl Rahtkens.

p. 770 (507) U. S. Pats. 409096 of Aug. 13, 1889 to Alois Blank; Reissue 11106 of Aug. 26, 1890 to Alois Blank; 1449058 of Mar. 20, 1923 to Thomas Robinson; 1559040 and 1559041 of Oct. 27, 1925 to J. H. Gillis; 1567048 of Dec. 29, 1925 to J. H. Gillis; 1574385 of Feb. 23, 1926 to J. H. Gillis; 1574446 of Feb. 23, 1926 to Thomas Robinson; 1589636 and 1589637 of Jun. 22, 1926 to J. H. Gillis; 1589841 of Jun. 22, 1926 to J. A. Daly; 1612885 of Jan. 4, 1927 to Thomas Robinson; 1625888 of Apr. 26, 1927 to J. H. Gillis; 1680096 of Aug. 7, 1927 to R. E. Drake; 1700774 of Feb. 5, 1929 to C. E. Rahr and R. E. Drake; 1701918 of Feb. 12, 1929 to J. H. Gillis; 1720708 of Jul. 16, 1929 to J. H. Young; 1753721 of Apr. 8, 1930 to Thomas Robinson; 1794449 of Mar. 3, 1931 to R. E. Drake; 1794748 of Mar. 3, 1931 to C. E. Yates; 1805920 of May 19, 1931 to Fred Muschler; 1852169 of Apr. 5, 1932 to Solomon Levy; 1871105 of Aug. 9, 1932 to C. E. Yates and H. C. Macan; 2042030 of May 26, 1936 to U. C. Tainton; Can. Pats. 39099 of Jun. 10, 1892 to R. G. Westphalen; 247312 of Mar. 3, 1925 to Anaconda Sales Co.; 253276 of Sep. 1, 1925 to J. H. Gillis; 267672 of Jan. 18, 1927 to Anaconda Sales Co.; 269401 of Mar. 29, 1927 to Anaconda Sales Co.; 278534 of Mar. 13, 1928 to Anaconda Sales Co.; 280921 of Jun. 12, 1928 to Anaconda Sales Co.; 312117 of Jun. 9, 1931 to Anaconda Sales Co.; 323649 of Jun. 28, 1932 to Anaconda Sales Co.; Brit. Pats. of 1889 (Mar. 27), 13329 to Alois Blank; 186363 of Mar. 29, 1921 to M. Höchstädter; 196063 of Jan. 12, 1922 to A. I. G. Warren; 199009 of May 15, 1923 to Roofing Research & Engineering Corp.; 211478 of Feb. 13, 1924 to Anaconda Sales Co.; 220647 of Aug. 15, 1924 to International Copperclad Co.; 278437 of Jul. 7, 1926 to A. I. G. Warren; 342487 of Jan. 15, 1929 to W. M. Shakespeare; 349690 of Mar. 22, 1930 to International Copperclad Co.; 368463 of Jan. 17, 1931 to International Copperclad Co.; 482064 of Mar. 23, 1938 to Union Minière du Haut Katanga; Ger. Pats. 43349 of May 25, 1888 to Arthur Siebel; 430641 of Feb. 10, 1924 to International Copperclad Co.; 489103 of Jul. 10, 1926 to Flintkote Co.; Appl. J-23.30 of Mar. 21, 1930 to International Copperclad Co.; Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.

p. 770 (508) U. S. Pats. 1488186 of Mar. 25, 1924 to J. H. Young; 1589841 of Jun. 22, 1926 to J. A. Daly; 1593205 of Jul. 20, 1926 to J. H. Young; 1720708 of Jul. 16, 1929 to J. H. Young; 1955572 of Apr. 17, 1934 to Jacob Adler and Paul Doerseln; Can. Pat. 349202 of Apr. 2, 1935 to Jacob Adler and Paul Doerseln; Brit. Pats. 190470 of Dec. 4, 1922 to H. H. Robertson Co.; 192377 and 192378 of Jan. 15, 1923 to H. H. Robertson Co.; Ger. Pat. 411273 of Jan. 18, 1923 to H. H. Robertson Co.

p. 770 (509) U. S. Pat. 1521128 of Dec. 30, 1924 to Thomas Robinson.

p. 770 (510) U. S. Pat. 2060083 of Nov. 10, 1936 to R. T. Johnston; Can. Pats. 362806 and 362807 of Dec. 22, 1936 to Bakelite Corp.

p. 770 (511) Can. Pat. 327877 of Nov. 22, 1932 to H. H. Robertson Co.

p. 770 (512) U. S. Pat. 845290 of Feb. 26, 1907 to E. H. Binns.

p. 770 (513) U. S. Pat. 2241819 of May 13, 1941 to Lester Kirschbraun and R. H. Cubberley.

p. 771 (514) U. S. Pats. 788358 of Apr. 25, 1905 to F. D. Jacobs; 816661 of Apr. 3, 1906 to F. D. Jacobs; Reissue 12475 of Apr. 24, 1906 to F. D. Jacobs; 1002301 of Sep. 5, 1911 to

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E. T. Newsome; 1053792 of Feb. 18, 1913 to J. F. Dietz; 1059682 of Apr. 22, 1913 to T. D. Miller; 1167949 of Jan. 11, 1916 to P. M. Stewart; Design 49171 of Jun. 13, 1916 to B. S. Annis; 1191932 of Jul. 18, 1916 to J. C. Loyer and O. M. Loyer; 1195090 of Aug. 15, 1916 to H. H. Robertson; 1226564 of May 15, 1917 to T. D. Miller; Reissue 14387 of Oct. 30, 1917 to J. C. Loyer and O. M. Loyer; 1277755 of Sep. 3, 1918 to H. H. Robertson; 1277758 of Sep. 3, 1918 to W. W. Roney; 1358113 of Nov. 9, 1920 to H. H. Robertson; 1471366 of Oct. 23, 1923 to A. A. Griswold; 1481559 of Mar. 11, 1924 to Frank Basel; 1516045 of Nov. 18, 1924 to Heinrich Kollbrunner; 1657979 of Jan. 31, 1928 to F. W. Thomas; 1700561 of Jan. 29, 1929 to F. J. Commin; 1814532 of Jul. 14, 1931 to W. A. Sutherland; 1868803 of Jul. 26, 1932 to F. C. Overbury; 1910312 of May 23, 1903 to Frank Young; Can. Pats. 185257 of Jul. 2, 1918 to T. D. Miller; 230268 of Apr. 10, 1923 to H. H. Robertson Co.; 233564 of Aug. 14, 1923 to H. H. Robertson Co.; 383048 of Aug. 1, 1939 to Barrett Co.; Brit. Pats. of 1912 (Jan. 17), 1360 to H. R. Wardell; 106997 and 106998 of Jun. 3, 1916 to H. H. Robertson Co.; 136984 of Mar. 24, 1919 to T. D. Miller; 159864 of Mar. 9, 1922 to H. H. Robertson Co.; 347105 of Jan. 10, 1920 to F. J. Commin and A. H. J. Wright; Ger. Pat. Design 743935 of May 15, 1920 to Chem. Fabriken Worms A.-G.

p. 771 (515) U. S. Pat. 1826706 of Oct. 13, 1931 to Isadore Davis.

p. 771 (516) U. S. Pats. 1358113 of Nov. 9, 1920 to H. H. Robertson; 1716706 of Jun. 11, 1929 to Ami Rey; 1740883 of Dec. 24, 1929 to H. A. Teeple; 2123247 of Jul. 12, 1938 to J. T. Lawrence and Leon Hartman; Can. Pat. 163058 of Jun. 8, 1915 to F. H. Grant.

p. 771 (517) U. S. Pats. 1765796 of Jun. 24, 1930 to Lester Kirschbraun; 2222939 of Nov. 26, 1940 to Bernard Feller; Brit. Pat. 353910 of Apr. 29, 1930 to Durastic Bituminous Products, Ltd.

p. 771 (518) U. S. Pats. 1730065 of Oct. 1, 1929 to A. C. Fischer; 1795913 of Mar. 10, 1931 to W. W. Weaver; 2080386 of May 11, 1937 to A. C. Fischer.

p. 771 (519) U. S. Pats. 394033 of Dec. 4, 1888 to S. E. Trott; 877019 of Jan. 21, 1908 to J. W. Troeger; 886912 of May 5, 1908 to C. W. Young and J. G. Burruss; 889235 of Jun. 2, 1908 to C. B. Jameson; 933221 of Sep. 7, 1909 to C. W. Young; 1048517 of Dec. 31, 1912 to J. G. Fox and H. W. Simms; 1104998 of Jul. 28, 1914 to F. C. Overbury; 1108884 of Sep. 1, 1914 to C. S. Bird; 1153418 of Sep. 14, 1915 to C. S. Bird; 1184509 of May 23, 1916 to C. S. Bird; 1447290 of Mar. 6, 1923 to A. C. Fischer; 1531151 of Mar. 24, 1925 to H. H. Steele; 1585692 and 1585693 of May 25, 1926 to Thomas Robinson; 1629287 of May 17, 1927 to S. K. Milligan; 1640906 of Aug. 30, 1927 to Thomas Robinson; 1644652 of Oct. 4, 1927 to Lester Kirschbraun; 1665450 of Apr. 10, 1928 to H. A. Garber; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; Reissue 17143 of Nov. 20, 1928 to A. C. Fischer; 1698242 of Jan. 8, 1929 to Thomas Robinson; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1716505 and 1716506 of Jun. 11, 1929 to Thomas Robinson; 1742724 of Jan. 7, 1930 to R. P. Perry; 1763601 of Jun. 10, 1930 to Lester Kirschbraun; 1776949 of Sep. 30, 1930 to P. E. Lombard.

p. 771 (520) U. S. Pats. 1261280 of Apr. 2, 1918 to F. C. Overbury; 1296324 of Mar. 4, 1919 to J. A. Scharwath; 1689985 of Oct. 30, 1928 to H. R. Wardell; 2270734 of Jan. 20, 1942 to Lester Kirschbraun; Can. Pats. 66636 of Dec. 19, 1905 to W. H. Wooswick; 190354 of May 13, 1919 to H. R. Wardell; 234293 of Sep. 18, 1923 to Steven Troy.

p. 771 (521) Can. Pat. 411810 of Apr. 13, 1943 to Patent & Licensing Corp.

p. 771 (522) U. S. Pat. 2316093 of Apr. 6, 1943 to A. D. MacNutt.

p. 771 (523) U. S. Pats. 322990 of Jul. 28, 1885 to Josef Sporny and Ian Zarski; 396292 of Jan. 15, 1889 to E. C. Richmond; 398332 of Feb. 19, 1889 to Julius Jaquet; 985140 of Feb. 28, 1911 to Hedley Button; 1208972 of Dec. 19, 1916 to Richard Jelier; 1403392 of Jan. 10, 1922 to Cecil Davis; 1438302 of Dec. 12, 1922 to Fred Frederiksen; 1480704 of Jan. 15, 1924 to H. A. Wheeler; 1536519 of May 5, 1925 to E. F. Parker; 1558005 of Oct. 20, 1925 to Dozier Finley; 1661562 of Mar. 6, 1928 to G. E. Conway; 2038192 of Apr. 21, 1936 to F. C. Overbury; 2062149 of Nov. 24, 1936 to G. R. Stark and J. A. Main; 2110579 of Mar. 8, 1938 to W. B. Robinson; 2256435 of Sep. 16, 1941 to C. W. Kraus; 2352116 of Jun. 20, 1944 to V. W. Noonan; Ger. Pat. Design 1260164 of Mar. 3, 1933 to Robert Beckmann.

p. 771 (524) U. S. Pats. 1448614 of Mar. 13, 1923 to A. H. Carrier; 1480704 of Jan. 15,

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1924 to H. A. Wheeler; 1953288 of Apr. 3, 1934 to M. L. Caton; 2039776 of May 5, 1936 to M. L. Caton; 2060084 of Nov. 10, 1936 to R. T. Johnston; 2253298 of Aug. 19, 1941 to H. J. Hyde; *Can. Pats.* 312298 of Jun. 16, 1931 to J. A. McKercher; 355646 of Jan. 28, 1936 to International Paper Co.

p. 771 (525) U. S. Pat. 1814291 of Jul. 14, 1931 to M. L. Caton; *Can. Pat.* 298017 of Mar. 4, 1930 to Flintkote Co.

p. 771 (526) U. S. Pats. 1088803 of Mar. 3, 1914 to G. F. Bishopric; 2130178 of Sep. 13, 1938 to Armin Elmendorf; *Can. Pat.* 363039 of Dec. 29, 1936 to Patent & Licensing Corp.

p. 771 (527) U. S. Pats. 1871090 of Aug. 9, 1932 to W. M. Shakespeare; 2026608 of Jan. 7, 1936 to S. S. Calafati; 2248723 of Jul. 8, 1941 to W. B. Robinson; 2253105 of Aug. 19, 1941 to B. H. Bill.

p. 771 (528) U. S. Pats. 2226239 of Dec. 24, 1940 to Armin Elmendorf; 2228362 of Jan. 14, 1941 to Robert Patterson.

p. 771 (529) U. S. Pat. 2279382 of Apr. 14, 1942 to G. E. Swenson.

p. 772 (530) U. S. Pats. 1887 of Dec. 10, 1840 to William Docker (slate); 55087 of May 29, 1866 to William Green; *Ger. Pats.* 707 of Jul. 4, 1877 to Ludwig Nagel; 100572 of Feb. 27, 1898 to Christian Fleisch; Design 1242227 of Apr. 4, 1932 to G. A. Türke.

p. 772 (531) U. S. Pat. 2132460 of Oct. 11, 1938 to H. A. Cumfer; *Can. Pats.* 297472 and 297473 of Feb. 11, 1930 to Guyton & Cumfer Mfg. Co.

p. 772 (532) U. S. Pats. 1497116 of Jun. 10, 1924 to C. M. Olson; 1783023 of Nov. 25, 1930 to S. G. Martin.

p. 772 (533) U. S. Pats. 292585 of Jan. 29, 1884 to E. B. Repp (metal); 1077095 of Oct. 28, 1913 to W. S. Orth; 1115866 of Nov. 3, 1914 to H. M. Reynolds; 1159766 of Nov. 9, 1915 to G. P. Heppes; 1172067 and 1172068 of Feb. 15, 1916 to A. S. Spiegel; 1192132 of Jul. 25, 1916 to A. S. Spiegel; 1227776 of May 29, 1917 to W. H. Garges; 1244654 of Oct. 30, 1917 to A. S. Spiegel; 1251704 of Jan. 1, 1918 to A. S. Spiegel; 1285147 of Nov. 19, 1918 to W. A. Harris; 1389979 of Sep. 6, 1921 to C. E. Rahr and Calvin Russell; 1417641 of May 30, 1922 to L. M. Stuffs; 1425991 and 1425992 of Aug. 15, 1922 to E. W. Leshner; 1442379 of Jan. 16, 1923 to Herbert Abraham; 1460827 of Jul. 3, 1923 to A. R. Purdy; 1474380 of Nov. 20, 1923 to Thomas Robinson; 1481193 of Jan. 15, 1924 to A. C. Fischer; 1528471 of Mar. 3, 1925 to C. H. Drumm; 1580755 and 1580756 of Apr. 13, 1926 to J. A. Murray; 1592014 and 1592015 of Jul. 13, 1926 to J. A. Topping; 1601127 of Sep. 28, 1926 to O. D. McFarland; 1602006 of Oct. 5, 1926 to A. C. Fischer; 1631936 of Jun. 7, 1927 to George Ritter; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; 1690396 of Nov. 6, 1928 to L. W. Block; 1719111 of Jul. 2, 1929 to R. A. Holdsworth; 1724269 of Aug. 13, 1929 to Dozier Finley; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; 1972028 of Aug. 28, 1934 to H. T. Nichols; 2064263 of Dec. 15, 1936 to S. A. Kiellar; *Can. Pats.* 152981 of Jan. 6, 1914 to W. S. Orth; 201056 of Jun. 15, 1920 to Flintkote Co.; 210450 of Apr. 12, 1921 to Roofing Patents Co.; 211667 of May 17, 1921 to Roofing Patents Co.; 216870 of Mar. 14, 1922 to Roofing Patents Co.; 220780 of Jul. 11, 1922 to Roofing Patents Co.; 220877 of Jul. 18, 1922 to C. E. Rahr; 223888 of Sep. 19, 1922 to Roofing Patents Co.; 224656 of Oct. 10, 1922 to Guyton & Cumfer Mfg. Co.; 244019 of Oct. 28, 1924 to Guyton & Cumfer Mfg. Co.; 274716 of Oct. 18, 1927 to P. E. Sinnett; 341267 of May 1, 1934 to Barrett Co.

p. 774 (534) U. S. Pats. 1295361 of Feb. 25, 1919 to F. C. Overbury; 1345099 of Jun. 29, 1920 to F. C. Overbury; 1366590 of Jan. 25, 1921 to F. C. Overbury; 1560301 of Nov. 3, 1925 to F. C. Overbury; *Can. Pat.* 248538 of Apr. 7, 1925 to Flintkote Co.

p. 774 (535) U. S. Pats. 1295361 of Feb. 25, 1919 to F. C. Overbury; 1365947 of Jan. 18, 1921 to F. C. Overbury; 1410903 of Mar. 28, 1922 to G. B. Ferguson; Reissue 15439 of Aug. 29, 1922 to G. B. Ferguson; 1467510 of Sep. 11, 1923 to G. T. Smith, Jr.; 1474380 of Nov. 20, 1923 to Thomas Robinson; 1476745 and 1476746 of Dec. 11, 1923 to E. J. Williams; 1542475 of Jun. 16, 1925 to R. A. Ruess; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; 2081018 of May 18, 1937 to T. L. Olson; *Can. Pat.* 321137 of Apr. 5, 1932 to C. S. Crary.

p. 774 (536) U. S. Pats. 1348498 of Aug. 3, 1920 to C. L. Keller; 1467779 of Sep. 11, 1923 to W. J. Dremann; 1516238 of Nov. 18, 1924 to C. W. Mortimer; 1536027 of Apr. 28, 1925

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to C. W. Mortimer; 1591042 of Jul. 6, 1926 to J. W. Ingels; 1614446 of Jan. 11, 1927 to A. R. Lukens; 1631936 of Jun. 7, 1927 to George Ritter; 1683285 of Sep. 4, 1928 to N. Z. Butterick; 1703170 of Feb. 26, 1929 to F. W. Preston; 1888055 of Nov. 15, 1932 to R. J. Tobin and G. A. Tobin; 1911141 of May 23, 1933 to A. C. Fischer; 1924650 of Aug. 29, 1933 to G. B. Payne; 1929165 of Oct. 3, 1933 to William Freegard; 2034602 of Mar. 17, 1936 to A. J. Anderson; 2066657 of Jan. 5, 1937 to J. B. Stevens; 2071430 of Feb. 23, 1937 to F. P. Reynolds; 2072289 of Mar. 2, 1937 to C. A. Barth; 2084981 of Jun. 29, 1937 to A. J. Anderson; 2087910 of Jul. 27, 1937 to H. H. Honigbaum; Design 109077 of Mar. 29, 1938 to T. W. Foley; 2151537 of Mar. 21, 1939 to J. B. Stevens; Can. Pats. 322046 of May 3, 1932 to Bird & Son, Inc.; 356255 of Mar. 3, 1936 to Bakelite Building Products Co., Inc.; 364281 of Feb. 23, 1937 to Building Products, Ltd.; 373232 of Apr. 19, 1938 to Johns-Manville Corp.; 373831 of May 17, 1938 to Lehon Co.

p. 774 (537) U. S. Pats. 310192 of Jan. 6, 1885 to J. T. Edson; 430362 of Jun. 17, 1890 to G. H. Babcock; 912057 of Feb. 9, 1909 to E. P. Auger (tile); 1070738 of Aug. 19, 1913 to Calvin Russell; 1115741 of Nov. 3, 1914 to Calvin Russell; 1116149 of Nov. 3, 1914 to A. S. Spiegel; Reissue 14058 of Feb. 8, 1916 to Calvin Russell; 1274605 of Aug. 6, 1918 to Calvin Russell; 1645767 of Oct. 18, 1927 to T. D. Miller; 1690908 of Nov. 6, 1928 to F. W. Preston; 2205679 of Jun. 25, 1940 to C. F. Ames, Jr.; Can. Pats. 155563 of May 12, 1914 to Calvin Russell; 220781 of Jul. 11, 1922 to Roofing Patents Co.; 374400 of Jun. 14, 1938 to Johns-Manville Corp.

p. 774 (538) U. S. Pats. 430366 of Jun. 17, 1890 to G. H. Babcock (tile); 1059682 of Apr. 22, 1913 to T. D. Miller (metal); 1076123 of Oct. 21, 1913 to O. F. Jacobson (metal); 1145440 of Jul. 6, 1915 to Calvin Russell; 1340348 of May 18, 1920 to C. E. Rahr; 1389979 of Sep. 6, 1921 to C. E. Rahr and Calvin Russell; 1496336 of Jun. 3, 1924 to M. P. Ennis; 1591042 of Jul. 6, 1926 to W. J. Ingels; 1592014 and 1592015 of Jul. 13, 1926 to J. A. Topping; 1685559 of Sep. 25, 1928 to W. R. Tobias; 1690396 of Nov. 6, 1928 to L. W. Block; 1691343 of Nov. 13, 1928 to J. B. French; 1698032 of Jan. 8, 1929 to G. R. Stark; 1699963 of Jan. 22, 1929 to H. R. French; 2013556 of Sep. 3, 1935 to C. R. Eckert; 2019701 of Nov. 5, 1935 to M. L. Hamlin; 2028004 of Jan. 14, 1936 to H. T. Nichols; 2047161 of Jul. 7, 1936 to Herbert Abraham; 2073274 of Mar. 9, 1937 to F. C. Young; 2103076 of Dec. 21, 1937 to N. P. Harshberger and K. M. Harshberger; Can. Pats. 166571 of Dec. 14, 1915 to Calvin Russell; 248541 of Apr. 7, 1915 to Flintkote Co.; 268273 of Feb. 8, 1927 to Flintkote Co.; 300414 of May 20, 1930 to Patent & Licensing Corp.; 319769 of Feb. 16, 1932 to H. T. Nichols; 333032 of Jun. 6, 1933 to Barrett Co.; 342751 of Jul. 3, 1934 to Barrett Co.

p. 774 (539) U. S. Pats. 1447290 of Mar. 6, 1923 to A. C. Fischer; 1488447 of Mar. 25, 1924 to J. A. Topping; 1552787 of Sep. 8, 1925 to R. M. Weston; 1580096 of Apr. 6, 1926 to F. J. Ballantine; 1592015 of Jul. 13, 1926 to J. A. Topping; 1648692 of Nov. 8, 1927 to J. A. McCarthy; Reissue 17143 of Nov. 20, 1928 to A. C. Fischer; 1717603 of Jun. 18, 1929 to N. P. Harshberger; 1725551 of Aug. 20, 1929 to F. E. Theilacker; Reissue 18698 of Dec. 20, 1932 to N. P. Harshberger; 2087911 of Jul. 27, 1937 to H. H. Honigbaum.

p. 774 (540) U. S. Pats. 595618 of Dec. 14, 1897 to Paul Gambs (slate); 1460795 of Jul. 3, 1923 to N. P. Harshberger (sides folded); 1479035 of Jan. 1, 1924 to A. C. Fischer; 1567538 of Dec. 29, 1925 to H. M. Morgan, Jr.; 1568807 of Jan. 5, 1926 to P. J. Dahm; 1582266 of Apr. 27, 1926 to N. P. Harshberger (sides folded); 1604745 of Oct. 26, 1926 to Dozier Finley; 1637306 of Jul. 26, 1927 to J. E. Hooker; 1641427 of Sep. 6, 1927 to N. P. Harshberger; 1657082 of Jan. 24, 1928 to R. P. Harshberger; 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1658806 of Feb. 14, 1928 to J. W. Kaufer; 1694418 of Dec. 11, 1928 to O. A. Heppes; 1696120 of Dec. 18, 1928 to J. E. Hooker; 1701704 of Feb. 12, 1929 to V. T. Stinson; 1717603 of Jun. 18, 1929 to N. P. Harshberger; 1756742 of Apr. 29, 1930 to N. P. Harshberger and R. P. Harshberger; 1806161 of May 19, 1931 to H. L. Guy; 1820388 of Aug. 25, 1931 to H. R. French; 1828222 of Oct. 20, 1931 to O. B. Clow; 1891229 of Dec. 20, 1932 to M. L. Hamlin; 1941105 of Dec. 26, 1933 to H. C. Naterman; 1962612 and 1962613 of Jun. 12, 1934 to G. B. Payne; 1974707 of Sep. 25, 1934 to M. A. F. Don; 1983936 of Dec. 11, 1934 to Dozier Finley; 1984529 of Dec. 18, 1934 to N. P. Harshberger; 2004879 of Jun. 11,

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1935 to E. C. Matthews and L. M. Clary; 2005335 of Jun. 18, 1935 to R. W. Clough; 2007710 of Jul. 9, 1935 to Dozier Finley; 2018722 of Oct. 19, 1935 to D. B. Humphrey; 2035369 and 2035370 of Mar. 24, 1936 to N. P. Harshberger; 2057245 of Oct. 13, 1936 to A. E. F. Moone; 2076014 of Apr. 6, 1937 to F. H. Bluhm; 2078998 of May 4, 1937 to E. R. Black; 2085552 of Jun. 29, 1937 to Trygve Storm; 2085553 of Jun. 29, 1937 to N. P. Harshberger; 2085554 of Jun. 29, 1937 to Harry Forbes; 2098488 of Nov. 9, 1937 to J. V. Donahue; 2100699 of Nov. 30, 1937 to J. H. Norrid; 2104067 of Jan. 4, 1938 to H. H. Bailey; 2104078 of Jan. 4, 1938 to M. L. Hamlin; 2117624 of May 17, 1938 to G. B. Payne; 2127695 of Aug. 23, 1938 to S. P. Miller; 2139015 of Dec. 6, 1938 to M. L. Hamlin; 2150883 of Mar. 14, 1939 to M. G. O'Reilly; 2158140 of May 16, 1939 to S. P. Miller; 2164712 of Jul. 4, 1939 to Lester Kirschbraun; 2168955 of Aug. 8, 1939 to J. A. Karan; 2173431 of Sep. 19, 1939 to F. L. Worsham; 2179738 of Nov. 14, 1939 to A. W. Fuller; 2182444 of Dec. 5, 1939 to R. C. McKinnie; Design 120622 of May 21, 1940 to F. M. Leslie; 2234446 of Mar. 11, 1941 to T. G. Murphy; 2242094 of May 13, 1941 to Vernon Tatro; 2247945 of Jul. 1, 1941 to E. H. Gardner; 2264083 of Nov. 25, 1941 to O. J. Kuhn; 2273879 of Feb. 24, 1942 to W. E. Maxey; *Can. Pats.* 185764 of Jul. 30, 1918 to W. H. Garges; 261699 of Jun. 15, 1926 to H. M. Morgan, Jr. (folded); 266411 of Dec. 7, 1926 to P. J. Dahm; 319848 of Feb. 16, 1932 to McHenry-Millhouse Mfg. Co.; 332852 of May 30, 1933 to Barrett Co.; 347155 and 347156 of Jan. 1, 1935 to Barrett Co.; 350917 of Jun. 11, 1935 to Patent & Licensing Corp.; 373487 and 373488 of May 3, 1938 to Barrett Co.; 375300 of Jul. 26, 1938 to Bakelite Building Products Co.; 381845 of Jun. 6, 1939 to Barrett Co.; 388838 of May 21, 1940 to Patent & Licensing Corp.; *Brit. Pats.* of 1903 (Feb. 5), 2765 to William Jackson; 121145 of Nov. 23, 1917 to Richard Fletcher and Harry Fletcher.

p. 774 (541) *U. S. Pats.* 1115866 of Nov. 3, 1914 to H. M. Reynolds; 1410867 of Mar. 28, 1922 to Herbert Abraham; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1488447 of Mar. 25, 1924 to J. A. Topping; 1500568 of Jul. 8, 1924 to Herbert Abraham; 1509795 of Sep. 23, 1924 to J. F. Tiner (metal); 1524125 of Jan. 27, 1925 to L. M. Ford; 1544956 of Jul. 7, 1925 to C. T. Torbert; 1642148 of Sep. 13, 1927 to H. L. Guy; 1664285 of Mar. 27, 1928 to R. J. Deans; 1685999 of Oct. 2, 1928 to W. A. Harris; 1729830 of Oct. 1, 1929 to N. P. Harshberger; 1787622 of Jan. 6, 1931 to H. G. Goslin; 1825576 of Sep. 29, 1931 to N. Z. Butterick; 1842761 of Jan. 26, 1932 to J. A. McCarthy; 1902155 of Mar. 21, 1933 to J. E. Berkheimer; 1943953 of Jan. 16, 1934 to J. V. Donahue; 2093944 of Sep. 21, 1937 to J. A. Topping; 2106395 of Jan. 25, 1938 to J. A. Topping; 2111798 of Mar. 22, 1938 to S. P. Miller and M. L. Hamlin; 2242094 of May 13, 1941 to Vernon Tatro; *Can. Pats.* 218497 of May 9, 1922 to Herbert Abraham; 233887 of Aug. 28, 1923 to Herbert Abraham; 236137 and 236138 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 333433 of Jun. 20, 1933 to Patent & Licensing Corp.; 358350 of Jun. 9, 1936 to J. A. Topping; 369897 of Nov. 9, 1937 to J. A. Topping; 376396 of Sep. 3, 1938 to Barrett Co.; *Brit. Pats.* 184361 of Jul. 26, 1921 to Herbert Abraham; 194700 of Mar. 10, 1922 to Herbert Abraham; 202921 of Feb. 27, 1923 to Herbert Abraham.

p. 774 (542) *U. S. Pats.* 1441420 of Jan. 9, 1923 to N. P. Harshberger; 1459827 of Jun. 26, 1923 to N. P. Harshberger; 1472270 of Oct. 30, 1923 to N. P. Harshberger; 1548017 of Aug. 4, 1925 to Herbert Abraham; 1584095 of May 11, 1926 to N. P. Harshberger; 1666203 of Apr. 17, 1928 to N. P. Harshberger; 1849785 of Mar. 15, 1932 to E. R. Black; 2050218 of Aug. 4, 1936 to Herbert Abraham; 2266376 of Dec. 16, 1941 to W. S. Miller; *Can. Pats.* 218133 of May 2, 1922 to N. P. Harshberger; 357587 of May 5, 1936 to Barrett Co.

p. 774 (543) *U. S. Pats.* 282439 of Jul. 31, 1883 to J. C. Chambers; 387360 of Aug. 7, 1888 to J. H. Keedy; 646623 of Apr. 3, 1900 to S. R. Hawthorne; 1153152 of Sep. 7, 1915 to Francis Brucker; 1270905 of Jul. 2, 1918 to H. W. White; 1322888 of Nov. 25, 1919 to A. C. Fischer; 1345683 of Jul. 6, 1920 to R. R. Mabie; 1463482 of Jul. 31, 1923 to W. J. Mountford, Jr.; 1468239 of Sep. 18, 1923 to B. C. Kridler; 1477167 of Dec. 11, 1923 to A. C. Fischer; 1493852 of May 13, 1924 to W. A. Fogg; 1498947 of Jun. 24, 1924 to J. O. Bewan; 1510497 of Oct. 7, 1924 to C. L. Keller; 1510756 of Oct. 7, 1924 to L. T. Ayrault and John Ayrault, Jr.; 1521893 of Jan. 6, 1925 to B. C. Kridler and J. C. Boyle; 1537952 of May 19, 1925 to B. C. Kridler and J. C. Boyle; 1544391 of Jun. 30, 1925 to V. J. Harward and W. P. Budd; 1561677

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of Nov. 17, 1925 to H. W. White; 1562409 of Nov. 17, 1925 to E. J. Brady; 1575662 of Mar. 9, 1926 to Frank Vandeven; 1579715 of Apr. 6, 1926 to B. C. Kridler and J. C. Boyle; 1582281 of Apr. 27, 1926 to B. C. Kridler and J. C. Boyle; 1613156 of Jan. 4, 1927 to J. C. Bergner; 1651392 of Dec. 6, 1927 to H. H. Honigbaum; 1692388 of Nov. 20, 1928 to W. A. Schollmeyer; 1725466 of Aug. 20, 1929 to P. S. MacMichael; 1750331 and 1750332 of Mar. 11, 1930 to W. A. Schollmeyer; 1790860 of Feb. 3, 1931 to A. L. Harvey; 2100254 of Nov. 23, 1937 to B. C. Kridler and P. W. Kridler; 2129833 of Sep. 13, 1938 to D. F. Fradette; 2138320 of Nov. 29, 1938 to H. F. Bozalina; 2227939 of Jan. 7, 1941 to B. C. Kridler; 2260446 of Oct. 28, 1941 to E. S. Fooks, Jr., and W. F. Koontz; *Can. Pats.* 48876 of May 8, 1895 to C. H. Dana; 166322 of Nov. 30, 1915 to Francis Brucker; 243654 of Oct. 14, 1924 to Richardson Co.; 262603 of Jul. 13, 1926 to Beaver Co., Ltd.; 370445 of Dec. 7, 1937 to B. C. Kridler; 375299 of Jul. 26, 1938 to Bakelite Building Products Co., Inc. [See also Reference p. 779 (562).]

p. 776 (544) U. S. Pats. 553514 of Jan. 28, 1896 to Frederick Crawford (metal); 1927436 of Sep. 19, 1933 to A. C. Fischer; 2085555 of Jun. 29, 1937 to O. J. Kuhn; 2089487, 2089488, 2089489, 2089490 of Aug. 10, 1937 to O. J. Kuhn; 2196420 of Apr. 9, 1940 to E. C. Matthews; *Can. Pat.* 49794 of Aug. 27, 1895 to Frederick Crawford.

p. 776 (545) U. S. Pats. 1685999 of Oct. 2, 1928 to W. A. Harris; 1860181 of May 24, 1932 to F. E. Horne; 2059233 of Nov. 3, 1936 to N. P. Harshberger; *Can. Pat.* 264815 of Oct. 5, 1926 to Flintkote Co.

p. 776 (546) U. S. Pats. Design 38931 of Dec. 3, 1907 to J. L. Dickelman; 1100955 of Jun. 23, 1914 to E. B. Coburn; 1104998 of Jul. 28, 1914 to F. C. Overbury; 1198653 of Sep. 19, 1916 to F. C. Overbury; 1301591 of Apr. 22, 1919 to F. C. Overbury; 1322220 of Nov. 18, 1919 to A. L. Baughman; 1547498 of Jul. 28, 1925 to G. M. Jordan; 1575974 of Mar. 9, 1926 to G. E. Conway; 1630019 of May 24, 1927 to F. P. Leonard; 1802868 of Apr. 28, 1931 to E. R. Black; 1819717 of Aug. 18, 1931 to C. L. Mangano; 1885346 of Nov. 1, 1932 to N. P. Harshberger; 1980760 of Nov. 13, 1934 to T. M. Mayfield; 2031993 of Feb. 25, 1936 to P. S. Varden; 2164636 of Jul. 4, 1939 to E. R. Black; 2253753 of Aug. 26, 1941 to E. R. Black; *Can. Pats.* 265985 of Nov. 23, 1926 to G. E. Conway; 315466 of Sep. 22, 1931 to Johns-Manville Corp.; 316830 of Nov. 3, 1931 to C. L. Mangano and R. M. Fleming.

p. 776 (547) U. S. Pats. 1076123 of Oct. 21, 1913 to O. F. Jacobson (metal); 1496336 of Jun. 3, 1924 to M. P. Ennis; 1582266 of Apr. 27, 1926 to N. P. Harshberger; 1667185 of Apr. 24, 1928 to W. E. Bartels; 1698032 of Jan. 8, 1929 to G. R. Stark; 2017230 of Oct. 15, 1935 to E. R. Black; 2164636 of Jul. 4, 1939 to E. R. Black.

p. 776 (548) U. S. Pats. 1385809 of Jul. 26, 1921 to Herbert Abraham; 1689290 of Oct. 30, 1928 to G. W. Mills, Jr.; *Can. Pat.* 214368 of Nov. 22, 1921 to Herbert Abraham (Ruberoid Co., Ltd.); *Brit. Pat.* 180150 of May 25, 1922 to Herbert Abraham.

p. 776 (549) U. S. Pats. 1438571 of Dec. 12, 1922 to Herbert Abraham; 1491015 of Apr. 22, 1924 to O. D. McFarland; 1604708 of Oct. 26, 1926 to G. W. Mills, Jr.; 2194427 of Mar. 19, 1940 to Lester Kirschbraun; 2197972 of Apr. 23, 1940 to A. F. Ernst; 2205679 of Jun. 25, 1940 to C. F. Ames, Jr.; 2212341 of Aug. 20, 1940 to C. R. Eckert; 2284705 of Jun. 2, 1942 to J. N. Wickersham; *Can. Pats.* 268272 and 268273 of Feb. 8, 1927 to Flintkote Co.; *Brit. Pat.* 219718 of Feb. 27, 1923 to Herbert Abraham.

p. 776 (550) U. S. Pats. 1119439 of Dec. 1, 1924 to R. B. Marschke; 2054917 of Sep. 22, 1936 to E. S. Yunik; *Can. Pat.* 266501 of Dec. 7, 1926 to Barrett Co.

p. 776 (551) U. S. Pats. 838232 of Dec. 11, 1906 to J. O. Ballentine; 1512248 of Oct. 21, 1924 to James Smith and M. E. Constable; 1513940 of Nov. 4, 1924 to James Smith and M. E. Constable; 1584054 of May 11, 1926 to H. R. Wardell; 1645767 of Oct. 18, 1927 to T. D. Miller; 1699213 of Jan. 15, 1929 to C. A. Statler; 1754771 of Apr. 15, 1930 to Lester Kirschbraun; 1759901 of May 27, 1930 to N. P. Harshberger; 1971932 of Aug. 28, 1934 to K. S. Guiterman (metal); 2171746 of Sep. 5, 1939 to K. S. Guiterman (metal); *Can. Pats.* 245173 of Dec. 9, 1924 to Johns-Manville Co.; 302453 of Jul. 29, 1930 to L. F. Lindley; 357757 of May 12, 1936 to Barrett Co.

p. 776 (552) U. S. Pats. 1201811 of Oct. 17, 1916 to H. H. Dupont; 1666088 of Apr. 17, 1928 to J. W. Farr; 1702414 and 1702415 of Feb. 19, 1929 to C. J. Richards; 1865771 of Jul.

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5, 1932 to Solomon Levy; 1983936 of Dec. 11, 1934 to Dozier Finley; 2080671 of May 18, 1937 to F. C. Overbury; 2081191 of May 25, 1937 to Lloyd Wright; *Can. Pat.* 370451 of Dec. 7, 1937 to F. C. Overbury; *Ger. Pat. Design* 331693 of Jan. 2, 1908 to Fr. Sieges & Sohn.

p. 776 (553) U. S. Pat. 1792543 of Feb. 17, 1931 to G. E. Livingston and W. H. C. Ness.

p. 776 (554) U. S. Pats. 1943258 of Jan. 9, 1934 to N. P. Harslberger; 2037297 of Apr. 14, 1936 to F. W. Yeager; 2065478 of Dec. 22, 1936 to Joseph Schulman.

p. 776 (555) U. S. Pats. 115912 of Jun. 13, 1871 to C. G. Van Pappelendam (metal); 154828 of Sep. 8, 1874 to Edwin Bennett (tile); 170582 of Nov. 30, 1875 to C. J. Merrill (tile); 211955 of Feb. 4, 1879 to Edwin Bennett (tile); 267904 of Nov. 21, 1882 to Lorenzo Lane and L. D. Woodworth (tile); Design 18208 of Mar. 27, 1888 to J. S. Thorn (metal); Design 20402 of Dec. 16, 1890 to G. H. Babcock (metal); 430365, 430369 and 430371 of Jun. 17, 1890 to G. H. Babcock (tile); Design 20635 of Mar. 24, 1891 to G. H. Babcock (metal); 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 595618 of Dec. 14, 1897 to Paul Gambs (slate); 605654 of Jun. 14, 1898 to Gustav Schulze (tile); Design 39274 of Apr. 21, 1908 to S. A. Jones (tile); 888825 of May 26, 1908 to H. E. Kock; 1070738 of Aug. 19, 1913 to Calvin Russell; 1772488 of Aug. 12, 1930 to B. E. Horne; 1799293 of Apr. 7, 1931 to H. R. French; 1928285 of Sep. 26, 1933 to A. C. Fischer; 2128836 of Aug. 30, 1938 to V. P. McVoy (metal); *Can. Pats.* 44903 of Dec. 18, 1893 to J. M. Olsen, H. P. Olsen and F. W. Bronn (tile); 306998 of Dec. 23, 1930 to Patent & Licensing Corp.; 416114 of Nov. 2, 1943 to Corning Glass Works (glass); *Ger. Pats.* 4940 of Aug. 2, 1878 to Hartwig Hüser; 6174 of Jul. 21, 1878 to Alex Brink (slate); *Norwegian Pat.* 9016 of Jul. 11, 1900 to C. J. Walls (cement).

p. 776 (556) U. S. Pats. 207989 of Sep. 10, 1878 to H. W. Shepard (metal); 267674 of Nov. 21, 1882 to Chester Comstock (metal); 292585 of Jan. 29, 1884 to E. B. Repp (metal); 294256 of Feb. 26, 1884 to L. H. Montross and J. C. West (metal); 309459 of Dec. 16, 1884 to M. F. Hamsley (metal); 341188 of May 4, 1886 to Ezekiel Van Noorden (metal); 482025 of Sep. 6, 1892 to E. B. Repp (metal); 562798 of Jun. 30, 1896 to Heinrich Bröcker (tile); 1515749 of Nov. 18, 1924 to N. G. Olsson (metal); 1566415 of Dec. 22, 1925 to Robert Miller (metal); 1637306 of Jul. 26, 1927 to J. E. Hooker; 1641427 of Sep. 6, 1927 to N. P. Harshberger; 1688612 of Oct. 23, 1928 to O. B. Clow; 1694418 of Dec. 11, 1928 to O. A. Heppes; 1696120 of Dec. 18, 1928 to J. E. Hooker; 1877222 of Sep. 13, 1932 to F. R. Brydle; 1968228 of Jul. 31, 1934 to Wright Smith, Jr.; 1983936 of Dec. 11, 1934 to Dozier Finley; 2068756 of Jan. 26, 1937 to S. P. Miller; *Can. Pats.* 260007 of Apr. 20, 1926 to Flintkote Co.; 301311 of Jun. 17, 1930 to J. E. Hooker; 367587 of Jul. 27, 1937 to Barrett Co.

p. 777 (557) U. S. Pats. 5091 of May 1, 1847 to Mathew Stewart (metal); 241805 of May 24, 1881 to W. G. Hyndman (metal); 790127 of May 16, 1905 to Ludwig Hatschek (tile); 1359167 of Nov. 16, 1920 to A. A. Griswold; 1430338 of Sep. 26, 1922 to J. A. Topping; Reissue 15556 of Mar. 6, 1923 to J. A. Topping; 1492516 of Apr. 29, 1924 to C. L. Keller; 1493374 of May 6, 1924 to W. J. Mountford, Jr.; 1496912 of Jun. 10, 1924 to J. A. Topping; 1508581 of Sep. 16, 1924 to John Shea; 1538235 of May 19, 1925 to A. L. Bell and F. A. Browne; 1552708 of Sep. 8, 1925 to W. A. Joy; 1557616 of Oct. 20, 1925 to W. J. Riley; 1561677 of Nov. 17, 1925 to H. W. White; 1566415 of Dec. 22, 1925 to Robert Miller; 1574345 of Feb. 23, 1926 to C. C. Gates; 1576423 of Mar. 9, 1926 to Jesse Fulenwider and Harry Fulenwider; 1582018 of Apr. 20, 1926 to J. C. Bergner; 1593407 and 1593408 of Jul. 20, 1926 to H. H. Honigbaum; 1618077 of Feb. 15, 1927 to Harry Fulenwider and Jesse Fulenwider; 1626780 of May 3, 1927 to W. G. Bickell; 1678804 of Jul. 31, 1928 to C. F. Ames, Jr.; 1681765 of Aug. 21, 1928 to C. C. Figue; 1686675 of Oct. 9, 1928 to C. F. Ames, Jr.; 1722962 of Jul. 30, 1929 to O. A. Heppes; 1730653 of Oct. 8, 1929 to G. G. Guertin; 1772487 and 1772488 of Aug. 12, 1930 to B. E. Horne; 1813798 of Jul. 7, 1931 to Anthony Gerosa; 1817743 of Aug. 4, 1931 to C. C. Figue; 1978841 of Oct. 30, 1934 to G. A. Holman; 2007855 of Jul. 9, 1935 to H. L. Guy; 2138320 of Nov. 29, 1938 to H. F. Bozalina; 2242094 of May 13, 1941 to Vernon Tatro; *Can. Pats.* 48037 of Jan. 28, 1895 to E. B. Repp (metal); 213089 of Aug. 23, 1921 to A. A. Griswold; 240231 of May 20, 1924 to J. A. Topping; 260006 of Apr. 20, 1926 to Flintkote Co.; *French Pat.* 455421 of Mar. 11, 1913 to Louis Bordat (cement).

p. 777 (558) U. S. Pats. 158123 of Dec. 22, 1874 to Joseph Ravoux (metal); 262475 of

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Aug. 8, 1882 to E. B. Repp (metal); 1436945 of Nov. 28, 1922 to R. K. Clifton; 1462772 of Jul. 24, 1923 to E. R. Snyder and C. W. Mortimer; 1666046 of Apr. 10, 1928 to O. A. Heppes; **Can. Pats.** 248798 of Apr. 21, 1925 to H. M. McLaughlin (metal); 260008 of Apr. 20, 1926 to Flintkote Co.

p. 777 (559) **U. S. Pats.** 1678333 of Jul. 24, 1928 to C. C. Figge; 1819717 of Aug. 18, 1931 to C. L. Mangano; 1820015 of Aug. 25, 1931 to C. C. Figge; 2017230 of Oct. 15, 1935 to E. R. Black; **Can. Pat.** 26965 of Jun. 16, 1887 to Nelson Green (metal).

p. 777 (560) **U. S. Pats.** 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 316134 of Apr. 24, 1885 to M. G. Farmer (metal); 341967 of May 18, 1886 to J. S. Thorn and Charles Lefflet (metal); Design 18209 of Mar. 27, 1888 to J. S. Thorn (metal); Design 19139 of Jun. 4, 1889 to L. L. Sagendorph (metal); 455271 and 455272 of Jun. 30, 1891 to Hermann Bormann; 516570 of Mar. 13, 1894 to Nicholas Monshausen (metal); 691650 of Jan. 21, 1902 to Nicholas Monshausen (metal); 1108236 of Aug. 25, 1914 to H. M. Reynolds; 1274605 of Aug. 6, 1918 to Calvin Russell; Design 56106 of Aug. 17, 1920 to Calvin Russell; 1604745 of Oct. 26, 1926 to Dozier Finley; 1675533 of Jul. 3, 1928 to H. A. Cumfer; 1679883 of Aug. 7, 1928 to A. S. Speer; 1691343 of Nov. 13, 1928 to J. B. French; 1699963 of Jan. 22, 1929 to H. R. French; 1744490 of Jan. 21, 1930 to C. W. Mortimer; 1775927 of Sep. 16, 1930 to Walter Becker; 1799293 of Apr. 7, 1931 to H. R. French; 1848965 of Mar. 8, 1932 to T. D. Miller; 1849779 of Mar. 15, 1932 to Harry Zimmerman; 1961896 of Jun. 5, 1934 to A. L. Barrall; Design 105124 of Jun. 29, 1937 to W. H. Molyneux; 2117094 of May 10, 1938 to H. B. Hutten; 2179738 of Nov. 14, 1939 to A. W. Fuller; **Can. Pats.** 23451 of Feb. 18, 1886 to J. B. Blaikie (slate); 260668 of May 11, 1926 to Barrett Co.; 285715 of Dec. 18, 1928 to Flintkote Co.; 286001 of Jan. 1, 1929 to J. B. French; 306998 of Dec. 23, 1930 to Patent & Licensing Corp.; 364890 of Mar. 16, 1937 to J. A. Topping; **Brit. Pat.** of 1867 (Mar. 17), 2612 to William Le Duc (slate); **Ger. Pat.** 182317 of Mar. 9, 1907 to H. L. Schwab (cement).

p. 777 (561) **U. S. Pats.** 124963 of Mar. 26, 1872 to Charles Lewando (metal); 207989 of Sep. 10, 1878 to H. W. Shepard (metal); 267674 of Nov. 21, 1882 to Chester Comstock (metal); 292585 of Jan. 29, 1884 to E. B. Repp (metal); 294256 of Feb. 26, 1884 to L. H. Montross and J. C. West (metal); 1431102 of Oct. 3, 1922 to J. F. Donahue and M. G. Staley; 1431103 of Oct. 3, 1922 to J. F. Donahue; 1469041 of Sep. 25, 1923 to H. L. Guy; 1486970 of Mar. 18, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1510534 and 1510535 of Oct. 7, 1924 to Herbert Abraham; 1558795 of Oct. 27, 1925 to J. F. Donahue; 1567817 of Dec. 29, 1925 to J. A. Scharwath; 1576090 of Mar. 9, 1926 to S. E. Carpenter; 1601735 of Oct. 5, 1926 to W. A. Harris; 1612700 of Dec. 28, 1926 to J. V. Cook; 1613099, 1613100 and 1613103 of Jan. 4, 1927 to N. P. Harshberger; 1648692 of Nov. 8, 1927 to J. A. McCarthy; 1658806 of Feb. 14, 1928 to J. W. Kaufer; 1663658 of Mar. 27, 1928 to N. P. Harshberger; 1669723 of May 15, 1928 to H. T. Nichols; 1669981 of May 15, 1928 to W. A. Harris; 1672713 of Jun. 5, 1928 to W. R. Durbin; 1674008 of Jun. 19, 1928 to H. L. Guy; 1688612 of Oct. 23, 1928 to O. B. Clow; 1696120 of Dec. 18, 1928 to J. E. Hooker; 1710954 of Apr. 30, 1929 to F. E. Theilacker; 1729830 of Oct. 1, 1929 to N. P. Harshberger; 1732229 of Oct. 22, 1929 to W. A. Harris; 1762404 and 1762405 of Jun. 10, 1930 to A. O. Mickelson and D. A. Cumfer; 1772487 of Aug. 12, 1930 to B. E. Horne; Reissue 17862 of Nov. 11, 1930 to W. A. Harris; Reissue 18515 of Jul. 12, 1932 to F. E. Theilacker; 1877222 of Sep. 13, 1932 to F. R. Brydle; 1889121 of Nov. 29, 1932 to N. P. Harshberger; 1893028 of Jan. 3, 1933 to H. H. Honigbaum; 1901161 of Mar. 14, 1933 to N. P. Harshberger; 1925239 of Sep. 5, 1933 to Harry Forbes; 1926070 of Sep. 12, 1933 to D. H. Sweet; 1934831 of Nov. 14, 1933 to D. H. Sweet; Reissue 19637 of Jul. 9, 1935 to N. P. Harshberger; 2013002 of Sep. 3, 1935 to G. S. Logan; 2055758 of Sep. 29, 1936 to J. A. Topping; 2086137 of Jul. 6, 1937 to George Ritter; 2081491 of May 25, 1937 to C. F. Martin; 2087911 of Jul. 25, 1937 to H. H. Honigbaum; 2184385 of Dec. 26, 1939 to O. C. Hall; 2194659 of Mar. 26, 1940 to B. H. Howle, Sr.; 2257152 of Sep. 30, 1941 to E. R. Black; **Can. Pats.** 229746 of Mar. 27, 1923 to J. F. Donahue and M. G. Staley; 229747 of Mar. 27, 1923 to J. F. Donahue; 236342 of Dec. 11, 1923 to H. L. Guy; 238434 of Mar. 11, 1924 to J. F. Donahue; 249097 of Apr. 28, 1925 to O. A. Heppes; 253786 of Sep. 22, 1925 to A. O. Mickelson and D. A. Cumfer; 255977 of Dec. 1, 1925 to Ford Roofing Prods. Co.; 259447 of Mar. 30, 1926 to H. L.

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Guy and C. S. Purnell; 263976 of Aug. 31, 1926 to Flintkote Co.; 267703 of Jan. 18, 1927 to Flintkote Co.; 269330 of Mar. 29, 1927 to N. P. Harshberger; 279378 of Apr. 17, 1928 to N. P. Harshberger; 283989 of Oct. 16, 1928 to H. H. Honigbaum; 301311 of Jun. 17, 1930 to J. E. Hooker; 309864 of Mar. 31, 1931 to Barrett Co.; 312125 of Jun. 9, 1931 to Barrett Co.; 342750 of Jul. 3, 1934 to Barrett Co.; 352314 of Aug. 13, 1935 to Black Systems, Inc.; 354389 of Nov. 26, 1935 to Barrett Co.; 363191 of Jan. 5, 1937 to Lehon Co.; 384410 of Oct. 3, 1939 to Barrett Co.

p. 779 (562) U. S. Pats. 1520947 of Dec. 30, 1924 to C. H. Harris; 1532421 of Apr. 7, 1925 to Hugh MacInnes and W. J. Riley; 1540960 of Jun. 9, 1925 to Roy Sherman; 1547469 of Jul. 28, 1925 to J. A. Topping; 1556649 of Oct. 13, 1925 to J. A. Topping; 1557392 of Oct. 13, 1925 to Herbert Abraham; 1557616 of Oct. 20, 1925 to W. J. Riley; 1560276 of Nov. 3, 1925 to Hugh MacInnes; 1570222 of Jan. 19, 1926 to J. C. Barley; 1582727 of Apr. 27, 1926, to J. J. Bradfield; 1612718 of Dec. 28, 1926 to J. F. Grice; 1626780 of May 3, 1927 to W. G. Bickell; 1659903 of Feb. 21, 1928 to P. C. Wolf; 1668291 of May 1, 1928 to J. A. Topping; 1722962 of Jul. 30, 1929 to O. A. Heppes; 1730347 of Oct. 8, 1929 to C. F. Ames, Jr. and C. W. Mortimer; 1748327 of Feb. 25, 1930 to E. R. Black; 1753583 of Apr. 8, 1930 to A. S. Speer; 1873944 of Aug. 23, 1932 to J. E. Black; 1962197 of Jun. 12, 1934 to H. H. Honigbaum; 2007855 of Jul. 9, 1935 to H. L. Guy; 2041780 of May 26, 1936 to W. L. Rowe; 2138464 of Nov. 29, 1938 to A. B. Walton; Can. Pats. 260006 of Apr. 20, 1926 to Flintkote Co.; 266290 of Nov. 30, 1926 to Beaver Co., Ltd.; 286851 of Jan. 29, 1929 to Lehon Co.; 390514 of Aug. 6, 1940 to Ford Roofing Products Co.; French Pat. 402181 of Sep. 30, 1909 to Carl Streckfuss (cement).

p. 779 (563) U. S. Pats. 124963 of Mar. 26, 1872 to Charles Lewando (metal); 140928 of Jul. 15, 1873 to Charles Lewando (metal); 158123 of Dec. 22, 1874 to Joseph Ravoux; 262475 of Aug. 8, 1882 to E. B. Repp (metal); 326374 of Sep. 15, 1885 to B. B. Adams; 406024 of Jul. 2, 1889 to J. D. Burton (metal); 740842 of Oct. 6, 1903 to Albert Friedley (metal); 1186619 of Jun. 13, 1916 to L. F. Tiefel and J. B. Tiefel (metal); 1427154 of Aug. 29, 1922 to A. A. Griswold; 1431476 of Oct. 10, 1922 to Hugh MacInnes; 1434116 of Oct. 31, 1922 to A. J. Hauber; 1436813 of Nov. 28, 1922 to Hugh MacInnes; 1436945 of Nov. 28, 1922 to R. K. Clifton; 1483882 of Feb. 19, 1924 to W. F. Harvey; 1508789 of Sep. 16, 1924 to C. H. Harris; 1529530 of Mar. 10, 1925 to R. L. Wyatt; 1533923 of Apr. 14, 1925 to R. A. Knoll; 1539716 of May 26, 1925 to W. T. Conley; 1570516 of Jan. 19, 1926 to T. D. Miller; 1588304 of Jun. 8, 1926 to A. L. Brownrigg; 1589719 of Jun. 22, 1926 to George Ritter; 1601735 of Oct. 5, 1926 to W. A. Harris; 1614871 of Jan. 18, 1927 to F. R. Brydle; 1625308 of Apr. 19, 1927 to William Freegard; 1630019 of May 24, 1927 to F. P. Leonard; 1634972 of Jul. 5, 1927 to N. R. Bartlett; 1635733 of Jul. 12, 1927 to G. R. Wyman; 1636349 of Jul. 19, 1927 to R. B. Adams; 1640806 of Aug. 30, 1927 to George Ritter; 1659575 of Feb. 21, 1928 to George Ritter; 1666046 of Apr. 10, 1928 to O. A. Heppes; 1669981 of May 15, 1928 to W. A. Harris; 1671424 of May 29, 1928 to N. P. Harshberger; 1697519 of Jan. 1, 1929 to W. W. Weaver; 1731187 of Oct. 8, 1929 to R. B. Adams; 1732229 of Oct. 22, 1929 to W. A. Harris; 1762404 and 1762405 of Jun. 10, 1930 to A. O. Mickelson and D. A. Cumfer; 1772487 of Aug. 12, 1930 to B. E. Horne; 1774861 of Sep. 2, 1930 to J. L. Wettlaufer; 1782416 of Nov. 25, 1930 to William Freegard; 2142996 of Jan. 10, 1939 to Ernest Bussey (metal); 2167192 of Jul. 25, 1939 to W. C. Weber (metal); 2184655 of Dec. 26, 1939 to R. L. Spain; Can. Pats. 220118, 220119 and 220120 of Jun. 27, 1922 to A. A. Griswold and J. F. Chevalier; 241178 of Jul. 1, 1924 to W. F. Harvey; 260008 of Apr. 20, 1926 to Flintkote Co.; 263976 of Aug. 31, 1926 to Flintkote Co.; 267703 and 267704 of Jan. 18, 1927 to Flintkote Co.; 272312 of Jul. 12, 1927 to Building Products Co., Ltd.; 278448 of Mar. 13, 1928 to R. H. Adams; 302350 of Jul. 22, 1930 to Patent & Licensing Corp.; 356256 of Mar. 3, 1936 to Bakelite Building Products Co., Inc.

p. 779 (564) U. S. Pats. 326374 of Sep. 15, 1885 to B. B. Adams; 1274410 of Aug. 6, 1918 to A. A. Griswold; 1279684 of Sep. 24, 1918 to A. A. Griswold; 1320136 of Oct. 28, 1919 to A. A. Griswold; 1321623 and 1321624 of Nov. 11, 1919 to A. A. Griswold; 1359167 of Nov. 16, 1920 to A. A. Griswold; 1427732 of Aug. 29, 1922 to A. A. Griswold; 1447019 of Feb. 27, 1923 to A. A. Griswold; 1450182 of Apr. 3, 1923 to Anthony Lamm; 1470837 of Oct. 16, 1923 to W. J. Hofstatter; 1483046 of Feb. 5, 1924 to George Ritter; 1483882 of Feb. 19, 1924 to

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W. F. Harvey; 1484020 of Feb. 19, 1924 to A. A. Griswold; 1498356 of Jun. 17, 1924 to Isadore Davis; 1508789 of Sep. 16, 1924 to C. H. Harris; 1512400 of Oct. 21, 1924 to F. R. Brydle; 1513800 of Nov. 4, 1924 to F. R. Brydle; 1522234 of Jan. 6, 1925 to William Freegard and M. S. Darrow; 1524432 of Jan. 27, 1925 to A. A. Griswold; 1548367 of Aug. 4, 1925 to F. E. Lietz; 1568750 of Jan. 5, 1926 to F. E. Lietz; 1571582 of Feb. 2, 1926 to A. A. Griswold and A. C. Goddard; 1582804 of Apr. 27, 1926 to J. W. Towler; 1583254 of May 4, 1926 to F. E. Lietz; 1589719 of Jun. 22, 1926 to George Ritter; 1593551 of Jul. 20, 1926 to Charles O'Dell; 1607455 of Nov. 16, 1926 to A. A. Griswold and A. C. Goddard; 1608738 of Nov. 30, 1926 to H. P. Hayden and W. W. Weaver; 1623127 of Apr. 5, 1927 to A. E. F. Moore; 1623474 of Apr. 5, 1927 to W. H. Hale; 1629558 of May 24, 1927 to H. R. Wardell; 1646551 of Oct. 25, 1927 to A. E. F. Moore; Reissue 16832 of Dec. 27, 1927 to Charles O'Dell; 1661067 of Feb. 28, 1928 to Harry Fulenwider and Jesse Fulenwider; 1666939 of Apr. 24, 1928 to J. F. Lietz; 1678804 of Jul. 31, 1928 to C. F. Ames, Jr.; 1684479 of Sep. 18, 1928 to C. R. Eckert; Reissue 17133 of Nov. 13, 1928 to F. E. Lietz; 1703156 of Feb. 26, 1929 to F. E. Lietz; 1744656 of Jan. 21, 1930 to C. J. McDavitt; 1757351 of May 6, 1930 to F. W. Yeager and W. P. Schulz; 1765058 of Jun. 17, 1930 to M. S. Darrow, W. J. Hart, F. G. Gronemeyer and F. B. Watkins; 1772924 of Aug. 12, 1930 to J. H. Weller; Reissue 17957 of Feb. 10, 1931 to J. F. Lietz; 1834966 of Dec. 8, 1931 to F. C. Overbury; 1842564 of Jan. 26, 1932 to F. C. Overbury; 1850088 of Mar. 22, 1932 to F. L. O. Wadsworth; 1895038 of Jan. 24, 1933 to J. O. Jones; 1927696 of Sep. 19, 1933 to Addison Applegate; 1940936 of Dec. 26, 1933 to E. R. Black; 1959519 of May 22, 1934 to E. R. Black; 1980760 of Nov. 13, 1934 to T. M. Mayfield; 2164636 of Jul. 4, 1939 to E. R. Black; 2201442 of May 21, 1940 to R. R. Mabie, Jr.; 2248336 of Jul. 8, 1941 to Ernest Bussey (metal); 2273220 of Feb. 17, 1942 to Alexander Ritter; *Can. Pats.* 24703 of Aug. 10, 1886 to B. B. Adams; 195071 of Dec. 16, 1919 to A. A. Griswold and J. F. Chevalier; 202333 of Jul. 27, 1920 to A. A. Griswold and J. F. Chevalier; 213089 of Aug. 23, 1921 to A. A. Griswold and J. F. Chevalier; 220117 of Jun. 27, 1922 to A. A. Griswold and J. F. Chevalier; 229746 and 229747 of Mar. 27, 1923 to J. F. Donahue; 231125 of May 15, 1923 to A. A. Griswold; 240310 of May 20, 1924 to C. H. Harris; 240467 of May 27, 1924 to Latite Shingle Corp.; 258942 of Mar. 16, 1926 to F. E. Lietz; 271758 of Jun. 21, 1927 to Barrett Co.; 273843 of Sep. 13, 1927 to Barber Asphalt Co.; 282018 of Jul. 24, 1928 to F. E. Lietz and J. F. Lietz; 310684 of Apr. 21, 1931 to Lehon Co.; 404788 of May 19, 1942 to R. R. Mabie, Jr.; *Brit. Pats.* 144699 of Apr. 25, 1918 to A. A. Griswold; 174268 of Jan. 26, 1922 to A. A. Griswold and J. F. Chevalier; *Ger. Pat.* 41774 of Mar. 31, 1887 to W. T. Westen.

p. 779 (565) U. S. Pat. 2008575 of Jul. 16, 1935 to Ernest Bussey; *Can. Pats.* 359770 of Aug. 11, 1936 to Starite Co.; 368952 of Sep. 28, 1937 to Certaineed Products Corp.

p. 779 (566) U. S. Pats. Design 1467 of Aug. 13, 1861 to Daniel Rickerby (slate); 121063 of Nov. 21, 1871 to O. J. Pierce (metal); 141215 of Jul. 29, 1873 to W. J. Fryer, Jr. and G. H. Johnson (slate); Design 19139 of Jun. 4, 1889 to L. L. Sagendorph (metal); Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; *Ger. Pat.* 22587 of Jun. 2, 1883 to Robert Spahlmann (tile).

p. 779 (567) U. S. Pats. 1450731 of Apr. 3, 1923 to R. S. Maclean; 1510534 of Oct. 7, 1924 to Herbert Abraham; 1576090 of Mar. 9, 1926 to S. E. Carpenter; 1595080 of Aug. 10, 1926 to A. C. Fischer; 1612700 of Dec. 28, 1926 to J. V. Cook; 1920474 of Aug. 1, 1933 to A. F. Martin; 1934666 of Nov. 7, 1933 to H. G. Goslin; Design 95242 of Apr. 16, 1935 to H. G. Goslin.

p. 779 (568) U. S. Pat. 1987133 of Jan. 8, 1935 to M. C. Smith.

p. 779 (569) U. S. Pats. Design 19888 of Jun. 10, 1890 to G. H. Babcock (metal); 1795277 of Mar. 3, 1931 to A. C. Fischer.

p. 779 (570) U. S. Pat. 320979 of Jun. 30, 1885 to L. B. Snow.

p. 779 (571) U. S. Pats. 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 316134 of Apr. 21, 1885 to M. G. Farmer (metal); 430362 and 430364 of Jun. 17, 1890 to G. H. Babcock (metal); Design 24084 of Mar. 5, 1895 to W. H. Mullins (metal); 888825 of May 26, 1908 to H. E. Kock; 954019 of Apr. 5, 1910 to O. O. Burnett (cement); 1799293 of Apr. 7, 1931 to H. R. French; *Can. Pat.* 306998 of Dec. 23, 1930 to Patent & Licensing Corp.

- p. 779 (572) U. S. Pats. 1441420 of Jan. 9, 1923 to N. P. Harshberger; 1604745 of Oct. 26, 1926 to Dozier Finley; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1729830 of Oct. 1, 1929 to N. P. Harshberger; Reissue 18772 of Mar. 21, 1933 to N. P. Harshberger; 2059234 of Nov. 3, 1936 to N. P. Harshberger; Can. Pat. 340686 of Apr. 10, 1934 to E. J. Hetzel.
- p. 779 (573) U. S. Pats. 1584739 of May 18, 1926 to Isadore Davis; 2143166 of Jan. 10, 1939 to A. W. Pattiani (metal); Can. Pat. 388837 of May 21, 1940 to Pateit & Licensing Corp.
- p. 779 (574) U. S. Pat. Design 77218 of Dec. 18, 1928 to A. J. Cron.
- p. 779 (575) U. S. Pats. 320979 of Jun. 30, 1885 to L. B. Snow; 1719777 of Dec. 3, 1929 to George Ritter.
- p. 779 (576) U. S. Pats. 1550693 of Aug. 25, 1925 to H. G. Goslin; 1596680 of Aug. 17, 1926 to W. E. Nelson; 1629287 of May 17, 1927 to S. K. Milligan; 165271 of Jan. 24, 1928 to W. E. Nelson; 1701640 of Feb. 12, 1929 to F. E. Sherriff; Design 91744 of Mar. 13, 1934 to J. A. Topping; Design 93642 of Oct. 16, 1934 to J. A. Topping; 2106396 of Jan. 25, 1938 to J. A. Topping.
- p. 780 (577) U. S. Pats. 1492610 of May 6, 1924 to J. T. Simpson; 1783839 of Dec. 2, 1930 to Henri Gauvin; Can. Pat. 306211 of Dec. 2, 1930 to Henri Gauvin.
- p. 780 (578) U. S. Pats. 1320502 of Nov. 4, 1919 to J. T. Simpson; 1492609 and 1492610 of May 6, 1924 to J. T. Simpson.
- p. 780 (579) U. S. Pat. 2175226 of Oct. 10, 1939 to Games Sayter; Can. Pat. 388560 of May 7, 1940 to Owens-Corning Fiberglas Corp.
- p. 780 (580) U. S. Pats. 1070738 of Aug. 19, 1913 to Calvin Russell; 1116149 of Nov. 3, 1914 to A. S. Spiegel; Reissue 14058 of Feb. 8, 1916 to Calvin Russell; 1274605 of Aug. 6, 1918 to Calvin Russell.
- p. 780 (581) U. S. Pat. 1699963 of Jan. 22, 1929 to H. R. French; Can. Pat. 285715 of Dec. 18, 1928 to Flintkote Co.
- p. 780 (582) U. S. Pats. Design 1467 of Aug. 13, 1861 to Daniel Rickerby (metal); 141215 of Jul. 29, 1873 to W. J. Fryer, Jr. and G. H. Johnson (slate); 1787622 of Jan. 6, 1931 to H. G. Goslin.
- p. 780 (583) U. S. Pats. 121063 of Nov. 21, 1871 to O. J. Pierce (slate); 316134 of Apr. 21, 1885 to M. G. Farmer (metal); 888825 of May 26, 1908 to H. E. Kock.
- p. 780 (584) U. S. Pat. 1690908 of Nov. 6, 1928 to F. W. Preston.
- p. 780 (585) U. S. Pat. 954019 of Apr. 5, 1910 to O. O. Burnett (cement); Can. Pat. 354135 of Nov. 12, 1935 to United Shoe Machinery Corp.
- p. 781 (586) U. S. Pats. 309433 of Dec. 16, 1884 to Charles Baillie; 414427 of Nov. 5, 1889 to C. A. Miller; 512986 of Jan. 16, 1894 to L. H. Montross; 908125 of Dec. 29, 1908 to F. C. Overbury; 1077095 of Oct. 28, 1913 to W. S. Orth; 1107762 of Aug. 18, 1914 to H. A. Cumfer; 1110238 of Sep. 8, 1914 to A. S. Spiegel; 1119439 of Dec. 1, 1914 to R. B. Marschke; 1150298 of Aug. 17, 1915 to F. C. Overbury; 1182416 and 1182417 of May 9, 1916 to F. C. Overbury; 1191297 of Jul. 18, 1916 to P. G. Gardner; 1192132 of Jul. 25, 1916 to A. S. Spiegel; 1249544 of Dec. 11, 1917 to A. S. Spiegel; 1260088 of Mar. 19, 1918 to A. S. Spiegel; 1261280 of Apr. 2, 1918 to F. C. Overbury; 1263987 of Apr. 23, 1918 to G. R. Wyman; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1301332 of Apr. 22, 1919 to A. S. Spiegel; 1301964 of Apr. 29, 1919 to F. C. Overbury; 1310082 of Jul. 15, 1919 to H. G. Hose; 1314476 and 1314477 of Aug. 26, 1919 to F. C. Overbury; 1340402 of May 18, 1920 to F. C. Overbury; 1358113 of Nov. 9, 1920 to H. H. Robertson; 1394342 of Oct. 18, 1921 to F. C. Overbury; 1394911 of Oct. 25, 1921 to R. D. King; 1410018 of Mar. 21, 1922 to O. P. Kiracofe; Reissue 15328 of Apr. 4, 1922 to F. C. Overbury; 1431869 of Oct. 10, 1922 to A. R. Bergen; 1441359 of Jan. 9, 1923 to S. M. Langston; 1441708 of Jan. 9, 1923 to F. C. Overbury; 1455342 of May 15, 1923 to S. M. Langston; 1462138 of Jul. 17, 1923 to S. M. Langston; 1464309 of Aug. 7, 1923 to H. A. Cumfer; 1473377 of Nov. 6, 1923 to S. M. Langston; 1478998 of Jan. 1, 1924 to W. R. Howard and W. H. Gregg; 1487661, 1487662 and 1487663 of Mar. 18, 1924 to S. M. Langston; 1489135 of Apr. 1, 1924 to S. M. Langston; 1489890 of Apr. 8, 1924 to S. M. Langston; 1501162 of Jul. 15, 1924 to H. A. Cumfer; 1515530 of Nov. 11, 1924 to P. P. Welty; 1532538 of Apr. 7, 1925 to S. M. Langston; 1551052 of Aug. 25, 1925 to R. T. Pollock; 1563245 of Nov. 24, 1925 to P. P. Welty; 1567920 of Dec. 29, 1925 to H. A. Cumfer; 1581236 of Apr.

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20, 1926 to A. S. Speer; 1593594 of Jul. 27, 1926 to R. P. Perry; 1599512 of Sep. 14, 1926 to W. H. Cady; 160128 of Sep. 28, 1926 to O. D. McFarland; 1601731 of Oct. 5, 1926 to John Flood; 1627665 of May 10, 1927 to F. C. Overbury; 1665450 of Apr. 10, 1928 to H. A. Garber; 1669999 of May 11, 1928 to C. M. Olsen; 1687873 of Oct. 16, 1928 to O. D. McFarland; Reissue 17187 of Jan. 8, 1929 to R. P. Perry; 1751945 of Mar. 25, 1930 to F. C. Overbury; 1762330 of Jun. 10, 1930 to H. R. Fielder and Amandus Grau; 1777076 of Sep. 30, 1930 to H. A. Cumfer; 1779216 of Oct. 28, 1930 to R. F. Paup; 1788122 of Jan. 6, 1931 to E. S. Petersen; 1816329 of Jul. 28, 1931 to R. T. Johnston; 1831422 of Nov. 10, 1931 to E. C. Otis; 1834004 of Dec. 1, 1931 to F. C. Overbury and O. A. Heppes; 1973863 of Sep. 18, 1934 to D. C. Betjeman; 1984647 of Dec. 18, 1934 to A. G. Leonard, Jr.; 2069444 of Feb. 2, 1937 to H. H. Honigbaum; *Can. Pat.* 152981 of Jan. 6, 1914 to W. S. Orth; 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 201057 and 201058 of Jun. 15, 1920 to Flintkote Co.; 211668 and 211669 of May 17, 1921 to Roofing Patents Co.; 214178 of Nov. 8, 1921 to Roofing Patents Co.; 216538 of Mar. 7, 1922 to Roofing Patents Co.; 223888 of Sep. 19, 1922 to Roofing Patents Co.; 239433 of Apr. 11, 1924 to Guyton & Cumfer Mfg. Co.; 247334 of Mar. 3, 1925 to Flintkote Co.; 253523 of Sep. 8, 1925 to Guyton & Cumfer Mfg. Co.; 254365 of Oct. 6, 1925 to P. P. Welty; 259399 of Mar. 30, 1926 to Flintkote Co.; 263204 of Aug. 3, 1926 to Flintkote Co.; 266926 of Dec. 21, 1926 to Flintkote Co.; 268578 of Feb. 22, 1927 to Flintkote Co.; 294016 of Oct. 15, 1929 to Flintkote Co.; 303385 of Aug. 26, 1930 to Barrett Co.; 312825 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co.; 314865 of Sep. 1, 1931 to Patent & Licensing Corp.; 330051 of Feb. 7, 1913 to Patent & Licensing Corp.; 358757 of Jun. 30, 1936 to J. A. Topping; *Brit. Pats.* 138874, 138883 and 138892 of Dec. 28, 1917 to Flintkote Co.; 162424 of May 5, 1921 to Flintkote Co.; *Ger. Pats.* 24619 of Mar. 13, 1883 to Hermann Schulz; 226401 of Dec. 25, 1907 to Flintkote Mfg. Co.; *French Pat.* 517127 of Jun. 14, 1920 to Flintkote Co.

p. 781 (587) U. S. Pats. 1265105 of May 7, 1918 to F. C. Overbury; Designs 53086 and 53087 of Mar. 11, 1919 to F. C. Overbury; 2188208 of Jan. 23, 1940 to W. M. Relue, Jr.

p. 781 (588) *Can. Pat.* 411540 of Aug. 17, 1943 to Allied Chemical & Dye Corp.

p. 781 (589) U. S. Pats. 1277861 of Sep. 3, 1918 to C. M. Clarke; 1298535 and 1298536 of Mar. 25, 1919 to E. A. Mastick, Jr.; 1559109 of Oct. 27, 1925 to Lester Kirschbraun; 1596449 of Aug. 17, 1926 to C. E. Rahr; *Can. Pats.* 205281 and 205282 of Nov. 2, 1920 to E. A. Mastick, Jr.

p. 781 (590) U. S. Pats. 1747631 of Feb. 4, 1930 to A. O. Mickelson; 1777076 of Sep. 30, 1930 to H. A. Cumfer; *Brit. Pat.* 352815 of Sep. 20, 1930 to H. A. Cumfer and W. J. Mason.

p. 781 (591) U. S. Pats. 1207513 of Dec. 5, 1916 to S. M. Ford; 1213472 of Jan. 23, 1917 to S. M. Ford; 1356500 of Oct. 19, 1920 to Henry Wulff; 1381388 of Jun. 14, 1921 to Henry Wulff; 1424459 of Aug. 1, 1922 to S. M. Ford; Reissues 15565 and 15566 of Mar. 27, 1923 to Henry Wulff; 1457662 and 1457663 of Jun. 5, 1923 to S. M. Ford; 1463314 of Jul. 31, 1923 to S. M. Ford; 1498555 of Jun. 24, 1924 to H. J. Langan; 1517582 of Dec. 2, 1924 to C. E. Rahr; 1646963 of Oct. 25, 1927 to H. H. Honigbaum; 1840984 of Jan. 12, 1932 to C. G. Talbott; 1911141 of May 23, 1933 to A. C. Fischer; 2106067 of Jan. 18, 1938 to F. H. Schmidt; *Can. Pats.* 179757 and 179758 of Oct. 16, 1917 to S. M. Ford; 204021 of Sep. 14, 1920 to S. M. Ford; 208684 of Feb. 22, 1921 to S. M. Ford; 248998 of Apr. 28, 1925 to H. J. Langan; 269633 of Apr. 5, 1927 to Flintkote Co.; 279907 of May 8, 1928 to H. H. Honigbaum.

p. 781 (592) U. S. Pats. 1704058 of Mar. 5, 1929 to F. C. Overbury; 1759995 of May 27, 1930 to F. C. Overbury; *Can. Pat.* 261769 of Jun. 15, 1906 to Flintkote Co.

p. 781 (593) U. S. Pat. 1843370 of Feb. 2, 1932 to F. C. Overbury.

p. 781 (594) U. S. Pats. 1318238 of Oct. 7, 1919 to A. S. Speer; 1379727 of May 31, 1921 to A. S. Speer; Reissue 15496 of Nov. 21, 1922 to A. S. Speer; Reissues 16390 and 16391 of Jul. 20, 1926 to A. S. Speer; *Can. Pat.* 217363 of Mar. 28, 1922 to Roofing Patents Co.

p. 781 (595) U. S. Pats. 1285147 of Nov. 19, 1918 to W. A. Harris; 1447567 of Mar. 6, 1923 to F. C. Overbury; 1513365 of Oct. 28, 1924 to G. C. Blohm; 1724269 of Aug. 13, 1929 to Dozier Finley; 1741566 of Dec. 31, 1929 to W. A. Harris.

p. 781 (596) U. S. Pats. 1531151 of Mar. 24, 1925 to H. H. Steele; 1563131 of Nov. 24,

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1925 to J. C. Whitmore; 1894614 and 1894615 of Jan. 17, 1933 to J. L. Wettlaufer; 2001399 of May 14, 1935 to J. A. Scharwath; 2249000 of Jul. 15, 1941 to R. T. Johnston; *Can. Pat.* 315859 of Oct. 6, 1931 to Black Systems, Inc.

p. 782 (597) U. S. Pats. 1325546 of Dec. 23, 1919 to H. R. Wardell; 1776949 of Sep. 30, 1930 to P. E. Lumbard; *Can. Pat.* 403745 of Mar. 31, 1942 to J. E. McNair.

p. 782 (598) U. S. Pats. 1345099 of Jun. 29, 1920 to F. C. Overbury; 1841296 of Jan. 12, 1932 to F. C. Overbury; *Can. Pat.* 330051 of Feb. 7, 1933 to Patent & Licensing Corp.

p. 782 (599) U. S. Pats. 1433983 of Oct. 31, 1922 to Frank Christenson; 1574098 of Feb. 23, 1926 to B. C. Kridler; 1729212 of Sep. 24, 1929 to A. C. Fischer; 1824471 and 1824472 of Sep. 22, 1931 to A. C. Fischer; 1879378 of Sep. 27, 1932 to W. C. McWilliams; 1898990 of Feb. 21, 1933 to N. P. Harshberger; 1928285 of Sep. 26, 1933 to A. C. Fischer; 1936327 of Nov. 21, 1933 to A. C. Fischer; 2250432 of Jul. 22, 1941 to J. P. Wilson.

p. 782 (600) *Can. Pat.* 390836 of Aug. 20, 1940 to Johns-Manville Corp.

p. 782 (601) U. S. Pat. 232230 of Jun. 29, 1943 to Trush McAvoy.

p. 782 (602) U. S. Pats. 1433983 of Oct. 31, 1922 to Frank Christenson; 1516238 of Nov. 18, 1924 to C. W. Mortimer; 1536027 of Apr. 28, 1925 to C. W. Mortimer; *Brit. Pat.* 113514 of Mar. 9, 1917 to H. J. Palmer.

p. 782 (603) U. S. Pats. 891501 of Jun. 23, 1908 to F. C. Overbury; 1348498 of Aug. 3, 1920 to C. L. Keller; 1348503 of Aug. 3, 1920 to A. R. Lukens, Jr.; 1464492 of Aug. 14, 1923 to Leon Busha; 1487662 of Mar. 18, 1924 to S. M. Langston; 1508365 of Sep. 9, 1924 to A. R. Lukens, Jr. and C. L. Keller; 1602007 of Oct. 5, 1926 to A. C. Fischer; 1614446 of Jan. 11, 1927 to A. R. Lukens, Jr.; 1629146 of May 17, 1927 to Leon Busha; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1702609 of Feb. 19, 1929 to E. W. Leshner; 1862852 of Jun. 14, 1932 to N. P. Harshberger; Design 90115 of Jun. 13, 1933 to N. P. Harshberger; Designs 92379 and 92380 of May 29, 1934 to J. A. Topping; Design 93824 of Nov. 13, 1934 to N. P. Harshberger; Design 96547 of Aug. 13, 1935 to N. P. Harshberger; 2013391 of Sep. 3, 1935 to M. W. Searls; 2068118 of Jan. 19, 1937 to J. A. Topping; *Can. Pats.* 352106 of Aug. 6, 1935 to N. P. Harshberger; 354855 of Dec. 17, 1935 to J. A. Topping; 359910 of Aug. 18, 1936 to Patent & Licensing Corp.; 360875 of Sep. 29, 1936 to Edward Karfol; 363128 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.

p. 783 (604) U. S. Pats. 1486346 of Mar. 11, 1924 to Cicero Hoskins; 1574098 of Feb. 23, 1926 to B. C. Kridler.

p. 783 (605) U. S. Pat. 1591042 of Jul. 6, 1926 to J. W. Ingels.

p. 783 (606) U. S. Pats. 1496108 of Jun. 3, 1924 to T. H. Wilson and E. R. Maltby; 1500709 of Jul. 8, 1924 to C. L. Keller.

p. 783 (607) U. S. Pat. 1570152 of Jan. 19, 1926 to J. E. Hooker.

p. 783 (608) U. S. Pat. 2235212 of Mar. 18, 1941 to Louis Herscovitz.

p. 783 (609) U. S. Pats. 1479035 of Jan. 1, 1924 to A. C. Fischer; 1488447 of Mar. 25, 1924 to J. A. Topping; 1494788 of May 20, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1709776 of Apr. 16, 1929 to Abbott Coburn; 1820388 of Aug. 25, 1931 to H. R. French; 2060618 of Nov. 10, 1936 to H. H. Honigbaum.

p. 783 (610) U. S. Pats. 1544956 of Jul. 7, 1925 to C. T. Torbert; 1607357 of Nov. 16, 1926 to L. L. Martin and C. R. Mayfield; 1770693 of Jul. 15, 1930 to N. P. Harshberger; 1919148 of Jul. 18, 1933 to H. L. Guy.

p. 783 (611) U. S. Pats. 1494789 of May 20, 1924 to Herbert Abraham; 1548017 of Aug. 4, 1925 to Herbert Abraham; 1818850 of Aug. 11, 1931 to N. P. Harshberger.

p. 783 (612) U. S. Pats. 1446858 of Feb. 27, 1923 to Max Rachlin; 1480167 of Jan. 8, 1924 to B. C. Kridler and J. C. Boyle; 1483735 of Feb. 12, 1924 to B. C. Kridler and J. C. Boyle; 1493852 of May 13, 1924 to W. A. Fogg; 1494707 of May 20, 1924 to Max Rachlin; 1498947 of Jun. 24, 1924 to J. O. Bewan; 1510497 of Oct. 7, 1924 to C. L. Keller; 1510756 of Oct. 7, 1924 to L. T. Ayrault and John Ayrault, Jr.; 1538329 of May 19, 1925 to H. H. Honigbaum; 1570222 of Jan. 19, 1926 to J. C. Barley; 1623127 of Apr. 5, 1927 to A. E. F. Moore; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1744589 of Jan. 21, 1930 to H. N. Striewig; 1925939 of Sep. 5, 1933 to Irving Sherman; 2007855 of Jul. 9, 1935 to H. L. Guy; *Can. Pats.* 262603

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of Jul. 13, 1926 to Beaver Co., Ltd.; 286850 of Jan. 29, 1929 to Lehon Co.; 310683 of Apr. 21, 1931 to Lehon Co.

p. 783 (613) U. S. Pat. 1570152 of Jan. 19, 1926 to J. E. Hooker.

p. 783 (614) U. S. Pats. 1209955 of Dec. 26, 1916 to S. M. Ford; 1415175 of May 9, 1922 to H. G. Hose; 1597135 of Aug. 24, 1926 to Lester Wittenberg; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1705497 of Mar. 19, 1929 to F. C. Overbury; Can. Pats. 248536 of Apr. 7, 1925 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.

p. 783 (615) U. S. Pat. 1402361 of Jan. 3, 1922 to H. G. Hose.

p. 783 (616) U. S. Pats. 1198653 of Sep. 19, 1916 to F. C. Overbury; 1582266 of Apr. 27, 1926 to N. P. Harshberger; 1596272 of Aug. 17, 1926 to G. M. Jordan; 1959519 of May 22, 1934 to E. R. Black; 1975986 of Oct. 9, 1934 to S. C. Straub; Can. Pat. 244166 of Nov. 4, 1924 to Charles Southgate.

p. 783 (617) U. S. Pats. 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1681975 of Aug. 28, 1928 to Isadore Davis.

p. 784 (618) U. S. Pats. 1063710 of Jun. 3, 1913 to W. F. McKay; 1219652 of Mar. 20, 1917 to W. F. McKay; 1426497 of Aug. 22, 1922 to O. K. Outman; 1451369 of Apr. 10, 1923 to R. P. Perry; 1455713 of May 15, 1923 to D. A. Cumfer; 1478544 of Dec. 25, 1923 to R. W. Bird; 1501512 of Jul. 15, 1924 to O. A. Bigler; 1534165 of Apr. 21, 1925 to D. A. Cumfer; 1593594 of Jul. 27, 1926 to R. P. Perry; 1627665 of May 10, 1927 to F. C. Overbury; 1969074 of Aug. 7, 1934 to N. P. Harshberger; 2112898 of Apr. 5, 1938 to George Knapp; 2352087 of Jun. 20, 1944 to E. W. Ericson and R. E. Brauer; Can. Pats. 175819 of Mar. 20, 1917 to W. F. McKay; 248537 of Apr. 7, 1925 to Flintkote Co.; 249587 of May 12, 1925 to Flintkote Co.; 251519 of Jul. 7, 1925 to Flintkote Co.; 256458 of Dec. 22, 1925 to H. A. Cumfer; 367890 of Aug. 10, 1937 to Certainteed Products Corp.

p. 784 (619) U. S. Pats. 1114032 of Oct. 20, 1914 to J. R. Powell and B. G. Casler; 1415923 of May 16, 1922 to R. B. Crabbs; 1418456 of Jun. 6, 1922 to F. C. Overbury; 1659808 of Feb. 21, 1928 to M. L. Caton; 1697464 of Jan. 1, 1929 to W. T. Hofmann; 1773117 of Aug. 19, 1930 to C. E. Rahr; Can. Pats. 229986 of Apr. 3, 1923 to W. T. Hofmann; 244197 of Nov. 4, 1924 to Barrett Co.; 249353 of May 5, 1925 to Flintkote Co.; 329238 of Jan. 10, 1933 to Certainteed Products Corp.; 332651 of May 23, 1933 to Certainteed Products Corp.; 335778 of Sep. 19, 1933 to Certainteed Products Corp.

p. 784 (620) U. S. Pats. 1154334 of Sep. 21, 1915 to F. C. Overbury; 1264831 of Apr. 30, 1918 to W. F. McKay; 1296984 of Mar. 11, 1919 to O. D. McFarland; Reissue 15489 of Nov. 14, 1922 to O. D. McFarland; 1456224 of May 22, 1923 to A. E. Currier; 1650285 of Nov. 22, 1927 to L. F. Lindley; 1898989 of Feb. 21, 1933 to N. P. Harshberger; 2099571 and 2099572 of Nov. 16, 1937 to W. H. Outman; 2100830 of Nov. 30, 1937 to H. F. Altheide; 2161440 of Jun. 6, 1939 to E. E. Venrick; 2170534 of Aug. 22, 1939 to A. D. McNutt; 2178273 of Oct. 31, 1939 to Lester Wittenberg; 2196847 of Apr. 9, 1940 to F. J. Austin; 2197972 of Apr. 23, 1940 to A. F. Ernst; 2212341 of Aug. 20, 1940 to C. R. Eckert; Can. Pats. 223401 of Sep. 5, 1922 to Roofing Patents Co.; 370239 of Nov. 30, 1937 to Certainteed Products Corp.; 389725 of Jul. 2, 1940 to Certainteed Products Corp.; 399468 of Sep. 23, 1941 to Barrett Co.; 417639 of Jan. 11, 1944 to Canadian Gypsum Co., Ltd.; Brit. Pat. 138875 of Jan. 21, 1915 to Flintkote Co.

p. 784 (621) U. S. Pats. 1623189 of Apr. 5, 1927 to Lester Kirschbraun; 1673991 of Jun. 19, 1928 to F. C. Overbury; 1756742 of Apr. 29, 1930, to N. P. Harshberger and R. P. Harshberger; 1958568 of May 15, 1934 to H. E. Beckman; 2112194 of Mar. 22, 1938 to N. P. Harshberger; Can. Pat. 260003 of Apr. 20, 1926 to F. C. Overbury.

p. 784 (622) U. S. Pats. 1365902 of Jan. 18, 1921 to S. M. Ford; 1619600 of Mar. 1, 1927 to H. A. Cumfer; 1795913 of Mar. 10, 1931 to W. W. Weaver; 2064473 of Dec. 15, 1936 to R. A. Holdsworth; Can. Pats. 372886 and 372887 of Mar. 29, 1938 to Building Products, Ltd.

p. 784 (623) U. S. Pat. 1613101 of Jan. 4, 1927 to N. P. Harshberger.

p. 784 (624) U. S. Pats. 1398250 of Nov. 29, 1921 to E. J. Yetter; 1469555 of Oct. 2, 1923 to H. A. Cumfer; 1475595 of Nov. 27, 1923 to C. E. Rahr and R. T. Pollock; 1484761 of Feb. 26, 1924 to H. A. Cumfer; 1577860 of Dec. 2, 1924 to C. E. Rahr and R. T. Pollock; 1595243 of Aug. 10, 1926 to R. T. Pollock; 1602007 of Oct. 5, 1926 to A. C. Fischer; 1699213

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of Jan. 15, 1929 to C. A. Statler; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1779175 of Oct. 21, 1930 to L. F. Lindley; 1913836 of Jun. 13, 1933 to M. L. Hamlin; 1993086 of Mar. 5, 1935 to S. W. Chaffee; *Can. Pats.* 249587 of May 12, 1925 to Flintkote Co.; 251355 of Jul. 7, 1925 to H. A. Cumfer; 256458 of Dec. 22, 1925 to H. A. Cumfer; 263005 of Jul. 27, 1926 to Flintkote Co.

p. 784 (625) U. S. Pat. 1759901 of May 27, 1930 to N. P. Harshberger.

p. 784 (626) U. S. Pat. 1126114 of Jan. 26, 1914 to A. S. Spiegel.

p. 784 (627) See Reference p. 774 (535).

p. 784 (628) U. S. Pats. 1495070 of May 20, 1924 to Dozier Finley; 1840997 of Jan. 12, 1932 to F. W. Yeager; 1860899 of May 31, 1932 to T. D. Miller; 1870414 of Aug. 9, 1932 to H. L. Levin and F. H. Neher; 1937933 of Dec. 5, 1933 to F. W. Yeager; 1974047 of Sep. 18, 1934 to N. P. Harshberger; 2171010 of Aug. 29, 1939 to C. C. Schuetz and F. B. Burns; 2174098 of Sep. 26, 1939 to Oscar Stein; *Can. Pats.* 242873 of Sep. 9, 1924 to Dozier Finley; 314865 of Sep. 1, 1931 to Patent & Licensing Corp.; 320408 of Mar. 8, 1932 to Patent & Licensing Corp.; 323534 of Jun. 21, 1932 to Patent & Licensing Corp.; 337158 of Nov. 14, 1933 to Patent & Licensing Corp.; 346638 of Dec. 11, 1934 to Barrett Co.; 380178 of Mar. 21, 1939 to Canadian Gypsum Co., Ltd.; 418681 of Feb. 29, 1944 to Canadian Gypsum Co., Ltd.

p. 784 (629) U. S. Pats. 1221370 of Apr. 3, 1917 to F. C. Overbury and H. C. Platts; 1265226 of May 7, 1918 to E. A. Mastick; 1410299 of Mar. 21, 1922 to N. P. Harshberger; 1469543 of Oct. 2, 1923 to G. I. Strachan and J. L. Strachan; *Can. Pat.* 210451 of Apr. 12, 1921 to Roofing Patents Co.

p. 784 (630) U. S. Pats. 1994643 of Mar. 19, 1935 to N. P. Harshberger; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2024861 of Dec. 17, 1935 to H. H. Honigbaum; 2209271 of Jul. 23, 1940 to N. P. Harshberger; *Can. Pat.* 364943 of Mar. 23, 1930 to Certaineed Products Corp.

p. 785 (631) U. S. Pat. 2132209 of Oct. 4, 1938 to E. R. Greenlee.

p. 785 (632) U. S. Pats. 194766 of Sep. 4, 1877 to Chester Comstock (metal); 520370 of May 22, 1894 to W. H. Mullins (metal); 1243064 of Oct. 16, 1917 to O. A. Heppes; 1290555 of Jan. 7, 1919 to O. A. Heppes; 1317384 of Sep. 30, 1919 to F. C. Overbury; Design 54917 of Apr. 20, 1920 to O. A. Heppes; 1447567 of Mar. 6, 1923 to F. C. Overbury; 1473919 of Nov. 13, 1923 to Herbert Abraham; 1973863 of Sep. 18, 1934 to D. C. Betjeman; *Can. Pats.* 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 170312 of Jun. 20, 1916 to O. A. Heppes; 347985 of Feb. 5, 1935 to Bakelite Building Products Co.; *Swiss Pat.* 5035 of May 9, 1892 to Johann Eberli (wood).

p. 785 (633) *Can. Pat.* 347571 of Jan. 15, 1935 to Self-Locking Carton Co.

p. 785 (634) U. S. Pat. 1345627 of Jul. 6, 1920 to F. C. Overbury.

p. 785 (635) U. S. Pat. 1824471 of Sep. 22, 1931 to A. C. Fischer.

p. 785 (636) U. S. Pat. 2272032 of Feb. 3, 1942 to C. W. Brown.

p. 787 (637) U. S. Pats. 1658685 of Feb. 7, 1928 to J. A. McCarthy; 1851088 of Mar. 29, 1932 to J. H. Duncan; 1919148 of Jul. 18, 1933 to H. L. Guy.

p. 787 (638) U. S. Pat. 978334 of Dec. 13, 1911 to F. C. Overbury; *Ger. Pat.* 252191 of Nov. 16, 1910 to Flintkote Mfg. Co.

p. 787 (639) U. S. Pats. 1024808 of Apr. 30, 1912 to Heinrich Schwarz; 1108236 of Aug. 25, 1914 to H. M. Reynolds; 1126932 of Feb. 2, 1915 to Herbert Abraham; 1445161 of Feb. 13, 1923 to Jacob Ott; 1500709 of Jul. 8, 1924 to C. L. Keller; Design 68522 of Oct. 20, 1925 to C. W. Dohm; 1604339 of Oct. 26, 1926 to H. A. Cumfer; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1682921 of Sep. 4, 1928 to O. D. McFarland; 1687873 of Oct. 16, 1928 to O. D. McFarland; 1758410 of May 13, 1930 to Hugo Reichel; 1766244 of Jun. 24, 1930 to H. A. Cumfer; 1773352 of Aug. 19, 1930 to Dozier Finley; 1846635 of Feb. 23, 1932 to Dozier Finley; 1851088 of Mar. 29, 1932 to J. H. Duncan; 1973863 of Sep. 18, 1934 to D. C. Betjeman; 2006417 of Jul. 2, 1935 to J. A. Topping; 2087595 of Jul. 20, 1937 to H. G. Goslin; 2291850 of Aug. 4, 1942 to J. A. Topping; *Can. Pats.* 139981 of Apr. 23, 1912 to J. C. Searle (metal); 237758 of Feb. 12, 1924 to Jacob Ott; 248539 of Apr. 7, 1925 to Flintkote Co.; 249094 of Apr. 28, 1925 to Flintkote Co.; 263204 of Aug. 3, 1926 to Flintkote Co.; 300349 of May 20, 1930

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to Barrett Co.; 312824 of Jun. 30, 1931 to Guyton & Cumfer Mfg. Co.; 334298 of Jul. 25, 1933 to Barrett Co.; 352256 of Aug. 6, 1935 to J. A. Topping; Ger. Pats. 249986 of Aug. 30, 1910 to Naumann Schefftel; Design 1437146 of Mar. 19, 1938 to Paul Richter; Austrian Pats. 48947 of Jul. 10, 1911 to Heinrich Schwarz; 53358 of May 12, 1912 to Heinrich Schwarz.

p. 787 (640) U. S. Pats. 1318238 of Oct. 7, 1919 to A. S. Speer; 1352154 of Sep. 7, 1920 to A. S. Speer; 1379727 of May 31, 1921 to A. S. Speer; Reissue 15496 of Nov. 21, 1922 to A. S. Speer; 1473919 of Nov. 13, 1923 to Herbert Abraham; 1486346 of Mar. 11, 1924 to Cicero Hoskins; Reissue 15955 of Nov. 25, 1924 to A. S. Speer; Reissue 16390 of Jul. 20, 1926 to A. S. Speer; 1831374 of Nov. 10, 1931 to J. A. Topping; 2158357 of May 16, 1939 to C. R. Eckert; Can. Pats. 210253 of Apr. 12, 1921 to Herbert Abraham; 211666 of May 17, 1921 to Roofing Patents Co.; 217363 of Mar. 28, 1922 to Roofing Patents Co.; Brit. Pat. 169874 of Oct. 13, 1921 to Herbert Abraham.

p. 787 (641) U. S. Pats. 1546868 of Jul. 21, 1925 to H. A. Sjodahl; Design 87104 of Jun. 7, 1932 to C. R. Eckert; 1956381 of Apr. 24, 1934 to C. R. Eckert; 1974047 of Sep. 18, 1934 to N. P. Harshberger; Design 104095 of Apr. 13, 1937 to J. R. Fife; Can. Pats. 263205 of Aug. 3, 1926 to Flintkote Co.; 363128 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.

p. 787 (642) U. S. Pats. 1802378 of Apr. 28, 1931 to N. P. Harshberger; 1956285 of Apr. 24, 1934 to N. P. Harshberger; 2097546 of Nov. 2, 1937 to H. D. Brown.

p. 787 (643) U. S. Pats. 1655885 of Jan. 10, 1928 to Reid Adair; 1732403 of Oct. 22, 1929 to W. A. Harris and H. R. French; Design 81768 of Aug. 5, 1930 to J. A. Topping; Design 88995 of Jan. 10, 1933 to R. J. Tobin; Can. Pats. 278369 and 278370 of Mar. 6, 1928 to Flintkote Co.; 284974 of Nov. 20, 1928 to Flintkote Co.

p. 787 (644) U. S. Pat. Design 107209 of Nov. 30, 1937 to J. J. Batell.

p. 787 (645) U. S. Pats. 341188 of May 4, 1886 to Ezekiel Van Noorden (metal); 1453373 of May 1, 1923 to Herbert Abraham; 1509795 of Sep. 23, 1924 to J. F. Tiner (metal); 1524125 of Jan. 27, 1925 to L. M. Ford; 1587017 of Jun. 1, 1926 to Hugh MacInnes; 1613102 of Jan. 4, 1927 to N. P. Harshberger; 1656434 of Jan. 17, 1928 to August Gietz; 1670469 of May 22, 1928 to J. A. McCarthy; 1721242 of Jul. 16, 1929 to J. P. Wolff; 1729846 of Oct. 1, 1929 to F. E. Theilacker; 1756741 and 1756743 of Apr. 29, 1930 to N. P. Harshberger; 1770693 of Jul. 15, 1930 to N. P. Harshberger; Reissue 17836 of Oct. 21, 1930 to J. A. McCarthy; 1956732 of May 1, 1934 to W. P. Schulz; Can. Pat. 362432 of Dec. 8, 1936 to Barrett Co.; Brit. Pat. 203988 of Feb. 27, 1923 to Herbert Abraham.

p. 787 (646) U. S. Pats. 1463632 of Jul. 31, 1923 to R. S. Severns; 1570222 of Jan. 19, 1926 to J. C. Barley; 1574099 of Feb. 23, 1926 to B. C. Kridler; 1612718 of Dec. 28, 1926 to J. F. Grice; 1623127 of Apr. 5, 1927 to A. E. F. Moone; 1627429 of May 3, 1927 to Isadore Davis; 1634465 of Jul. 5, 1927 to H. G. Kelly; Design 75433 of Jun. 5, 1928 to H. G. Kelly; 1744589 of Jan. 21, 1930 to H. N. Strieweg; 1768134 of Jun. 24, 1930 to A. E. F. Moone; 1775937 of Sep. 16, 1930 to C. L. Keller; 1814405 of Jul. 14, 1931 to Alvin Myhre; 1980053 of Nov. 6, 1934 to N. P. Harshberger; 2037163 of Apr. 14, 1936 to H. L. Guy; 2080912 of May 18, 1937 to H. L. Guy; 2227939 of Jan. 7, 1941 to B. C. Kridler; 2272377 of Feb. 10, 1942 to G. S. Logan; Can. Pat. 310683 of Apr. 21, 1931 to Lehon Co.

p. 787 (647) U. S. Pats. 1402361 of Jan. 3, 1922 to H. G. Hose; 1453373 of May 1, 1923 to Herbert Abraham; 1559506 of Oct. 27, 1925 to William Fregard; 1619601 of Mar. 1, 1927 to H. A. Cumfer; 1659575 of Feb. 21, 1928 to George Ritter; 1677492 of Jul. 17, 1928 to George Ritter; 1731187 of Oct. 8, 1929 to R. B. Adams; 1771990 of Aug. 5, 1930 to J. C. Bergner; 1993163, 1993164, 1993165 and 1993166 of Mar. 5, 1935 to N. P. Harshberger; Can. Pats. 248535 of Apr. 7, 1923 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.; 311390 of May 19, 1931 to R. B. Adams; Brit. Pat. 203988 of Feb. 27, 1923 to Herbert Abraham.

p. 787 (648) U. S. Pats. 1681975 of Aug. 28, 1928 to Isadore Davis; 1685493 of Sep. 25, 1928 to H. G. Kelly; 1741515 of Dec. 31, 1929 to Martin Halprin; 2017230 of Oct. 15, 1935 to E. R. Black.

p. 787 (649) U. S. Pats. 1868751 of Jul. 26, 1932 to H. C. Koch; 1900861 of Mar. 7, 1933 to N. P. Harshberger; 1945485 of Jan. 30, 1934 to N. P. Harshberger; Reissue 19903

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of Mar. 24, 1936 to N. P. Harshberger; *Can. Pat.* 351714 of Jul. 16, 1935 to Patent & Licensing Corp.

p. 787 (650) *U. S. Pat.* 2100830 of Nov. 30, 1937 to H. F. Altheide.

p. 787 (651) *U. S. Pats.* 1602007 of Oct. 5, 1926 to A. C. Fischer; 1602164 of Oct. 5, 1926 to F. C. Overbury; 1619598 of Mar. 1, 1927 to H. A. Cumfer; 1619601 of Mar. 1, 1927 to H. A. Cumfer; 1627429 of May 3, 1927 to Isadore Davis; 1698891 of Jan. 15, 1929 to F. C. Overbury; 1802378 of Apr. 28, 1931 to N. P. Harshberger; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2041761 of May 26, 1936 to N. P. Harshberger; 2209271 of Jul. 23, 1940 to N. P. Harshberger; *Can. Pats.* 248535 of Apr. 7, 1925 to Flintkote Co.; 260005 of Apr. 20, 1926 to Flintkote Co.

p. 787 (652) *U. S. Pats.* 1949907 of Mar. 6, 1934 to N. P. Harshberger; Design 91654 of Mar. 6, 1934 to N. P. Harshberger.

p. 789 (653) *U. S. Pats.* 373373 of Nov. 15, 1867 to L. H. Montross (metal); 512986 of Jan. 16, 1894 to L. H. Montross (metal); 668625 of Feb. 26, 1901 to C. W. Connor (metal); 820294 of May 8, 1906 to Albert Friedley (metal); 1150298 of Aug. 17, 1915 to F. C. Overbury; 1340402 of May 18, 1920 to F. C. Overbury; 1583977 of May 11, 1926 to H. G. Kelly; 1776949 of Sep. 30, 1930 to P. E. Lumbard; 1834004 of Dec. 1, 1931 to F. C. Overbury and O. A. Heppes; *Can. Pats.* 69189 of Oct. 30, 1900 to C. W. Connor (metal); 165873 of Nov. 9, 1915 to Flintkote Mfg. Co.; 347571 of Jan. 15, 1935 to Self-Locking Carton Co.; *Ger. Pat.* Design 1437146 of Mar. 19, 1938 to Paul Richter.

p. 789 (654) *U. S. Pats.* 1326899 of Jan. 6, 1920 to Herbert Abraham; Reissue 15280 of Feb. 7, 1922 to Herbert Abraham; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1482776 of Feb. 5, 1924 to Herbert Abraham; 1548107 of Aug. 4, 1925 to E. T. Street; 1593594 of Jul. 27, 1926 to R. P. Perry; *Can. Pats.* 199466 of Apr. 27, 1920 to Herbert Abraham; 219560 of Jun. 13, 1922 to Ruberoid Co., Ltd.; 233887 of Aug. 28, 1923 to Ruberoid Co., Ltd.; 236139 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 242500 of Aug. 26, 1924 to Ruberoid Co., Ltd.; 285501 of Dec. 11, 1928 to Barrett Co.; 303385 of Aug. 26, 1930 to Barrett Co.; *Brit. Pat.* 152858 of Oct. 13, 1919 to Herbert Abraham.

p. 789 (655) *U. S. Pats.* 1412295 of Apr. 11, 1922 to A. S. Speer; 1455232 of May 15, 1923 to A. S. Speer; Reissue 16429 of Sep. 21, 1926 to A. S. Speer; 1623983 of Apr. 12, 1927 to A. S. Speer; *Can. Pat.* 127361 of Mar. 28, 1922 to Roofing Patents Co.

p. 789 (656) *U. S. Pats.* 1415758 of May 9, 1922 to Herbert Abraham; 1557391 of Oct. 13, 1925 to Herbert Abraham; 2158357 of May 16, 1939 to C. R. Eckert; *Can. Pats.* 219560 of Jun. 13, 1922 to Herbert Abraham; 233150 of Jul. 31, 1923 to Ruberoid Co., Ltd.; 242295 of Aug. 19, 1924 to Ruberoid Co., Ltd.

p. 789 (657) *U. S. Pat.* 1936327 of Nov. 21, 1933 to A. C. Fischer.

p. 790 (658) *U. S. Pats.* Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; 1464493 and 1464494 of Aug. 14, 1923 to Leon Busha; 1500709 of Jul. 8, 1924 to C. L. Keller; 1533969 of Apr. 14, 1925 to Leon Busha; Reissue 16621 of May 10, 1927 to Leon Busha; Design 89783 of May 2, 1933 to A. E. F. Moone; 1927436 of Sep. 19, 1933 to A. C. Fischer; *Can. Pat.* 259057 of Mar. 16, 1926 to W. E. Nelson.

p. 790 (659) *U. S. Pats.* 1442614 of Jan. 16, 1923 to J. E. Hooker; 1510533 of Oct. 7, 1924 to Herbert Abraham; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker; 1756741 of Apr. 29, 1930 to N. P. Harshberger and R. P. Harshberger.

p. 790 (660) *U. S. Pats.* 1494707 of May 20, 1924 to Max Rachlin; 1544392 of Jun. 30, 1925 to V. J. Harward and W. P. Budd; Design 75433 of Jun. 5, 1928 to H. G. Kelly.

p. 790 (661) *U. S. Pats.* 1659575 of Feb. 21, 1928 to George Ritter; 1795277 of Mar. 3, 1931 to A. C. Fischer.

p. 790 (662) *U. S. Pat.* 1885346 of Nov. 1, 1932 to N. P. Harshberger.

p. 790 (663) *U. S. Pats.* 1466077 of Aug. 28, 1923 to H. R. Wardell; 1584343 of May 11, 1926 to Herbert Abraham; *Can. Pats.* 236136 of Dec. 4, 1923 to Ruberoid Co., Ltd.; 261144 of May 25, 1926 to Johns-Manville, Inc.

p. 790 (664) *U. S. Pats.* 1619599 of Mar. 1, 1927 to H. A. Cumfer; 2024861 of Dec. 17, 1935 to H. H. Honigbaum.

- p. 790 (665) U. S. Pats. 346209 of Jul. 27, 1886 to Frederick Mankey (wood); 512986 of Jan. 16, 1894 to L. H. Montross (wood); 520370 of May 22, 1894 to W. H. Mullins (metal); Design 24082 of Mar. 5, 1895 to W. H. Mullins (metal); 1295360 of Feb. 25, 1919 to F. C. Overbury; Design 54160 of Nov. 4, 1919 to R. L. Woodruff; 1455232 of May 15, 1923 to A. S. Speer; Reissue 16429 of Sep. 21, 1926 to A. S. Speer; Design 74669 of Mar. 13, 1928 to J. R. Fife; 1974047 of Sep. 18, 1934 to N. P. Harshberger; Designs 99248 and 99249 of Apr. 7, 1936 to J. J. Piazza; Design 104971 of Jun. 15, 1937 to John Logan, Jr.; Can. Pats. 46251 of Jun. 5, 1894 to H. D. Walker (metal); 67812 of Jun. 21, 1900 to H. D. Walker (metal); 341469 of May 8, 1934 to Building Products, Ltd.; Ger. Pat. Design 1437146 of Mar. 19, 1938 to Paul Richter.
- p. 790 (666) U. S. Pats. 891501 of Jun. 23, 1908 to F. C. Overbury; 1274623 of Aug. 6, 1918 to A. S. Spiegel; 1301964 of Apr. 29, 1919 to F. C. Overbury; 1447567 of Mar. 6, 1923 to F. C. Overbury; Design 67565 of Jun. 16, 1925 to A. W. Abbey and C. W. Dohm; 1599253 of Sep. 7, 1926 to Max Skolnik; 1666204 of Apr. 17, 1928 to N. P. Harshberger; 1670942 of May 22, 1928 to Walter Thomsen; Design 75612 of Jun. 26, 1928 to L. C. Cotner; Design 81712 of Jul. 29, 1930 to A. E. F. Moore; 1773352 of Aug. 19, 1930 to Dozier Finley; Design 84668 of Jul. 14, 1931 to S. H. Ralph; Designs 85638 and 85639 of Dec. 1, 1931 to Dozier Finley; Design 86085 of Jan. 26, 1932 to H. L. Guy; 1846635 of Feb. 23, 1932 to Dozier Finley; Design 87057 of May 31, 1932 to H. L. Guy; Design 89639 of Apr. 18, 1933 to D. B. Humphrey; Design 89783 of May 2, 1933 to A. E. F. Moore; 1913768 of Jun. 13, 1933 to A. E. F. Moore; Design 92504 of Jun. 12, 1934 to J. A. Topping; Design 92632 of Jun. 26, 1934 to J. A. Topping; Design 93191 of Aug. 28, 1934 to J. A. Topping; 2006417 of Jul. 2, 1935 to J. A. Topping; 2018718 of Oct. 29, 1935 to N. P. Harshberger; Can. Pats. 205148 of Oct. 26, 1920 to Flintkote Co.; 220782 of Jul. 11, 1922 to Roofing Patents Co.; 352770 of Sep. 3, 1935 to Certainitee Products Corp.; 364727 of Mar. 16, 1937 to J. A. Topping; 365088 of Mar. 30, 1937 to Bakelite Building Products Co., Inc.; Austrian Pat. 48947 of Jul. 10, 1911 to Heinrich Schwarz; Swiss Pat. 5035 of May 9, 1892 to Johann Eberli (wood).
- p. 790 (667) U. S. Pats. 1641858 of Sep. 8, 1927 to J. A. McCarthy; 1705497 of Mar. 19, 1929 to F. C. Overbury; 1718933 of Jun. 25, 1929 to N. P. Harshberger; 1919148 of Jul. 18, 1933 to H. L. Guy.
- p. 790 (668) U. S. Pats. 742614 of Oct. 27, 1903 to J. L. M. Du Four; 1683016 of Sep. 4, 1928 to E. L. Bell and O. M. Beebe.
- p. 790 (669) U. S. Pat. 1345627 of Jul. 6, 1920 to F. C. Overbury.
- p. 790 (670) U. S. Pats. 1633474 of Jun. 21, 1927 to Leon Busha; 1688917 of Oct. 23, 1928 to Leon Busha; Design 76843 of Nov. 6, 1928 to J. A. Topping; 1812654 of Jun. 30, 1931 to Lester Kirschbraun; 1913836 of Jun. 13, 1933 to M. L. Hamlin; 1975487 of Oct. 2, 1934 to J. A. Topping; 2199760 of May 7, 1940 to C. C. Schuetz; Can. Pats. 330050 of Feb. 7, 1933 to Patent & Licensing Corp.; 355399 of Jan. 14, 1936 to J. A. Topping.
- p. 790 (671) U. S. Pats. Design 61363 of Aug. 15, 1922 to N. Z. Butterick; 1445991 of Feb. 20, 1923 to N. Z. Butterick; 1701640 of Feb. 12, 1929 to F. E. Sherriff; 1741539 of Dec. 31, 1929 to J. M. Richardson; 1812654 of Jun. 30, 1931 to Lester Kirschbraun; 1863178 of Jun. 14, 1932 to H. R. Wood; Design 105276 of Jul. 13, 1937 to A. G. Hauck; 2096968 of Oct. 26, 1937 to R. T. Johnston; Design 107312 of Dec. 7, 1937 to A. C. Fischer; Can. Pats. 232178 of Jun. 26, 1923 to N. Z. Butterick; 313515 of Jul. 21, 1931 to Patent & Licensing Corp.; Brit. Pat. 195097 of Mar. 20, 1922 to N. Z. Butterick.
- p. 790 (672) U. S. Pat. 1812424 of Jun. 30, 1931 to H. R. Wood.
- p. 790 (673) U. S. Pat. Design 83704 of Mar. 17, 1931 to G. R. Stark; Can. Pats. 368951 of Sep. 28, 1937 to Certainitee Products Corp.; 369792 of Nov. 9, 1937 to Certainitee Products Corp.
- p. 790 (674) U. S. Pats. Design 53015 of Feb. 18, 1919 to F. C. Overbury and H. C. Platts; 1442614 of Jan. 16, 1923 to J. E. Hooker; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker.
- p. 790 (675) U. S. Pats. Design 74467 of Feb. 14, 1928 to Herbert Abraham; Design 77213 of Dec. 18, 1928 to Herbert Abraham.
- p. 791 (676) U. S. Pats. 1555441 of Sep. 29, 1925 to H. A. Sjodahl; Design 68552 of Oct. 20, 1925 to H. A. Sjodahl; Designs 70988 and 70989 of Aug. 31, 1926 to H. A. Sjodahl; Designs

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75761 and 75762 of Jul. 17, 1928 to Herbert Abraham; 1768280 of Jun. 24, 1930 to Salvatore Arcidiacono; *Can. Pats.* 266300 of Nov. 30, 1926 to Chatfield Mfg. Co.; Designs 7620 and 7621 of Jun. 28, 1927 to H. A. Sjodahl.

p. 791 (677) U. S. Pat. Design 83718 of Mar. 24, 1931 to H. D. Brown.

p. 791 (678) U. S. Pat. 1843370 of Feb. 2, 1932 to F. C. Overbury.

p. 791 (679) U. S. Pats. 1442614 of Jan. 16, 1923 to J. E. Hooker; 1511732 of Oct. 14, 1924 to F. J. Kromenaker and G. M. Kromenaker; Designs 70986 and 70987 of Aug. 31, 1926 to H. A. Sjodahl; 1935656 of Nov. 21, 1933 to C. W. Mortimer; Design 101732 of Oct. 27, 1936 to O. B. Clow; *Can. Pats.* 267307 of Jan. 4, 1927 to Chatfield Mfg. Co.; Designs 7618 and 7619 of Jun. 28, 1927 to H. A. Sjodahl.

p. 791 (680) U. S. Pats. Design 86194 of Feb. 16, 1932 to H. D. Brown; Design 91061 of Nov. 21, 1933 to J. R. Fife; Design 92132 of May 1, 1934 to R. A. Holdsworth.

p. 791 (681) U. S. Pats. Design 89471 of Mar. 14, 1933 to R. S. Streeter and O. E. Hendrick; Design 104948 of Jun. 15, 1937 to A. O. Mickelson; 2088686 of Aug. 3, 1937 to B. W. Blanchard, Jr.; Design 135035 of Feb. 16, 1943 to D. B. Humphrey; Design 135045 of Feb. 16, 1943 to O. A. Bigler and M. S. Larrison.

p. 791 (682) U. S. Pats. Design 57126 of Feb. 22, 1921 to O. A. Heppes; 1475551 of Nov. 27, 1923 to F. C. Overbury; *Can. Pat.* 256566 of Dec. 22, 1925 to Flintkote Co.

p. 791 (683) U. S. Pats. 1489921 of Apr. 8, 1924 to T. J. Brown; 2037163 of Apr. 14, 1936 to H. L. Guy; 2144678 of Jan. 24, 1939 to F. P. Goldschmidt; *Can. Pats.* 256795 and 256796 of Dec. 29, 1925 to Flintkote Co.

p. 791 (684) U. S. Pat. 1257321 of Feb. 26, 1918 to H. A. Cumfer and O. D. McFarland.

p. 791 (685) U. S. Pat. 1745631 of Feb. 4, 1930 to A. O. Mickelson.

p. 791 (686) *Can. Pat.* 273858 of Sep. 13, 1927 to Flintkote Co.

p. 791 (687) U. S. Pats. 1326899 of Jan. 6, 1920, to Herbert Abraham; Reissue 15280 of Feb. 7, 1922 to Herbert Abraham; *Can. Pat.* 244166 of Nov. 4, 1924 to Charles Southgate.

p. 793 (688) U. S. Pats. 1157665 of Oct. 26, 1915 to M. B. Becker; 1174960 of Mar. 14, 1916 to M. B. Becker; 1181827 of May 2, 1916 to C. S. Bird; 1256508 of Feb. 19, 1918 to M. B. Becker; 1263987 of Apr. 23, 1918 to G. R. Wyman; 1270654 of Jan. 25, 1918 to F. C. Overbury; 1276881 of Aug. 27, 1918 to H. A. Cumfer and O. D. McFarland; 1295361 of Feb. 25, 1919 to F. C. Overbury; 1314476 and 1314477 of Aug. 26, 1919 to F. C. Overbury; 1368947 of Feb. 15, 1921 to W. W. Lewis; 1501512 of Jul. 15, 1924 to O. A. Bigler; 1515530 of Nov. 11, 1924 to P. P. Welty; 1546782 of Jul. 21, 1925 to L. M. Ford; 1563245 of Nov. 24, 1925 to P. P. Welty; 1599512 of Sep. 14, 1926 to W. H. Cady; 1612776 of Dec. 28, 1926 to Lester Kirschbraun; 1635676 of Jul. 12, 1927 to Louis Isaacs; 1722702 of Jul. 30, 1929 to Lester Kirschbraun; 1755049 of Apr. 15, 1930 to W. H. Cady; 1756989 of May 6, 1930 to F. C. Overbury; 1760873 of Jun. 3, 1930 to Lester Kirschbraun; 1767374 of Jun. 24, 1930 to Lester Kirschbraun; 1767401 of Jun. 24, 1930 to F. M. Reuter; 1781877 of Nov. 18, 1930 to H. L. Levin; 1783575 of Dec. 2, 1930 to C. R. MacDonald; 1783849 and 1783850 of Dec. 2, 1930 to C. R. MacDonald; 1807918 of Jun. 2, 1931 to Lester Kirschbraun and O. A. Heppes; Design 84808 of Aug. 11, 1931 to L. E. Calkins; 1826674 of Oct. 6, 1931 to B. H. Roberts and Erich Gach; 1843056 of Jan. 26, 1932 to P. P. Welty; 1860180 and 1860181 of May 24, 1932 to F. E. Horne; 1862627 of Jun. 14, 1932 to Robert Maclean; 1864806 of Jun. 28, 1932 to D. A. Cumfer; 1865579 of Jul. 5, 1932 to C. R. MacDonald; 1870426 of Aug. 9, 1932 to G. J. Snyder; 1872185 and 1872186 of Aug. 16, 1932 to R. W. B. Reade; 1873209 of Aug. 23, 1932 to C. R. MacDonald; 1873213 of Aug. 23, 1932 to F. C. Overbury; 1890017 of Dec. 6, 1932 to C. R. MacDonald; 1890018 of Dec. 6, 1932 to F. C. Overbury; 1905553 of Apr. 25, 1933 to R. D. Falk; 1908313 of May 9, 1933 to H. D. Brown; 1911014 of May 23, 1933 to C. P. Cowan; 1915964 of Jun. 27, 1933 to A. L. Wall; 1931554 of Oct. 24, 1933 to A. E. F. Moore; 1942449 of Jan. 9, 1934 to F. H. Rahr; 1943686 of Jan. 16, 1934 to O. V. McGrew; 1958571 and 1958572 of May 15, 1934 to F. H. Gilchrist; 1959519 of May 22, 1934 to E. R. Black; 1968535 of Jul. 31, 1934 to A. E. F. Moore; 1972810 of Sep. 4, 1934 to J. L. Wertlauber; 1975324 of Oct. 2, 1934 to G. P. Jordan; 1976662 of Oct. 9, 1934 to J. A. Feely; 1993134 of Mar. 5, 1935 to W. W. Ford; 1997381 of Apr. 9, 1935 to Albert Horowitz; Design 95250 of Apr. 16, 1935 to N. P. Harshberger;

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1999903 of Apr. 30, 1935 to N. P. Harshberger; 2000030 of May 7, 1935 to George Knapp; 2005462 of Jun. 18, 1935 to J. H. Gibson; 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2012709 of Aug. 27, 1935 to C. P. Cowan; 2013317 of Sep. 3, 1935 to B. H. Roberts and E. L. Chamberlain; 2027029 of Jan. 7, 1936 to C. R. Eckert; Design 99132 of Mar. 31, 1936 to R. S. Maclean; 2035871 of Mar. 31, 1936 to C. R. Eckert; 2038102 of Apr. 21, 1936 to C. R. Eckert; 2043959 of Jun. 9, 1936 to N. P. Harshberger; 2045423 of Jun. 23, 1936 to J. A. Topping; 2047741 and 2047742 of Jul. 14, 1936 to S. P. Miller; 2056521 of Oct. 6, 1936 to J. F. Honan; 2057545 of Oct. 13, 1936 to S. C. Straub; 2059490 of Nov. 3, 1936 to Antoinette Reisig; 2059520 of Nov. 3, 1936 to N. P. Harshberger; Design 101921 of Nov. 10, 1936 to R. S. Maclean; 2063935 of Dec. 15, 1936 to Lester Kirschbraun; 2085899 of Jul. 6, 1937 to E. L. Chamberlain; 2090490 of Aug. 17, 1937 to S. C. Straub; 2094688 of Oct. 5, 1937 to G. N. Wallace and F. C. McKinley; 2101589 of Dec. 7, 1937 to R. S. Maclean; 2110485 of Mar. 8, 1938 to J. B. Hunt; 2113303 of Apr. 5, 1938 to Alexis Kiefer; 2118250 of May 24, 1938 to George Knapp; 2118632 of May 24, 1938 to J. L. Wettlaufer; 2162886 of Jun. 20, 1939 to M. L. Hamlin; 2164508 of Jul. 4, 1939 to G. A. Fasold; 2169136 of Aug. 8, 1939 to R. R. Brenner; 2209283 of Jul. 23, 1940 to D. B. Ronzone (metal); Can. Pats. 185435 of Jul. 9, 1918 to F. C. Overbury; 254365 of Oct. 6, 1925 to P. P. Welty; 283901 of Oct. 9, 1928 to Building Products, Ltd.; 287870 of Mar. 12, 1929 to Building Products, Ltd.; 304113 of Sep. 23, 1930 to C. P. Cowan; 320760 of Mar. 22, 1932 to Bird & Son, Inc.; 320864 of Mar. 22, 1932 to Patent & Licensing Corp.; 323048 of Jun. 7, 1932 to Bird & Son, Inc.; 323447 of Jun. 21, 1932 to Bird & Son, Inc.; 328694 of Dec. 20, 1933 to Patent & Licensing Corp.; 333340 of Jun. 20, 1934 to Robert Maclean; 340998 of Apr. 17, 1934 to Building Products, Ltd.; 346329 of Nov. 27, 1934 to Barrett Co.; 346637 of Dec. 11, 1934 to Barrett Co.; 347157 of Jan. 1, 1935 to Barrett Co.; 350701 and 350702 of Jun. 4, 1935 to Barrett Co.; 354872 of Dec. 24, 1935 to J. A. Feely; 363127 of Jan. 5, 1937 to Bakelite Building Products Co., Inc.; 364944 of Mar. 23, 1937 to Certaineed Products Corp.; 365094 and 365095 of Mar. 30, 1937 to Certaineed Products Corp.; 365948 of May 4, 1937 to J. A. Topping; 368954 and 368955 of Sep. 28, 1937 to Certaineed Products Corp.; 369379 of Oct. 19, 1937 to Patent & Licensing Corp.; 385572 of Dec. 12, 1939 to J. B. Wardle (metallic fastener); 387697 of Mar. 26, 1940 to Charles Weir; 388026 of Apr. 16, 1940 to Barrett Co.; 389419 of Jun. 18, 1940 to Building Products, Ltd.

p. 793 (689) U. S. Pats. 466742 of Jan. 5, 1892 to D. N. Lanyon; 691434 of Jan. 21, 1902 to Morris Barnett; 703640 of Jul. 1, 1902 to G. P. Chappell; 735344 of Aug. 4, 1903 to W. N. Cornell; 852688 of May 7, 1907 to H. J. Wade; 957189 of May 10, 1910 to G. P. Chappell; 1107074 of Aug. 11, 1914 to W. M. Janpole; 1153512 of Sep. 14, 1915 to C. G. Muench; 1171081 of Feb. 8, 1916 to W. E. Aycock; 1202770 of Oct. 31, 1916 to W. E. Aycock; 1269906 of Jun. 18, 1918 to C. M. Clarke; 1278943 of Sep. 17, 1918 to W. V. Lander; 1317158 of Sep. 30, 1919 to G. H. Ellis; 1333628 of Mar. 16, 1920 to T. B. Munroe; 1376553 of May 3, 1921 to W. V. Lander; 1376587 and 1376588 of May 3, 1921 to J. K. Shaw; 1431125 and 1431126 of Oct. 3, 1922 to C. G. Robinson; 1449605 of Mar. 27, 1923 to Benedikt Holzmayer; 1503211 of Jul. 29, 1924 to J. K. Shaw; 1545212 of Jul. 7, 1925 to G. B. Stowe; 1572919 of Feb. 16, 1926 to C. D. Geese; 1604938 of Oct. 26, 1926 to William Goldie, Sr.; 1773695 of Aug. 19, 1930 to C. W. Morden; 1831058 of Nov. 10, 1931 to H. A. Cumfer; 1871090 of Aug. 9, 1932 to W. M. Shakespeare; 1871887 of Aug. 16, 1932 to Andrew Jasinski; 1941769 of Jan. 2, 1934 to G. J. Ward; 1958571 of May 15, 1934 to F. H. Gilchrist; 1972810 of Sep. 4, 1934 to J. L. Wettlaufer; 1986829 of Jan. 8, 1935 to J. A. Klimsza; 2018216 of Oct. 22, 1935 to R. S. Maclean; 2026608 of Jan. 7, 1936 to S. S. Calafati; Design 99132 of Mar. 31, 1936 to R. S. Maclean; Design 101921 of Nov. 10, 1936 to R. S. Maclean; 2063935 of Dec. 15, 1936 to Lester Kirschbraun; 2101589 of Dec. 7, 1937 to R. S. Maclean; 2114450 of Apr. 19, 1938 to R. S. Maclean; 2115172 of Apr. 26, 1938 to Lester Kirschbraun; 2122577 of Jul. 5, 1938 to L. H. Mattes and R. S. Maclean; 2128824 of Aug. 30, 1938 to J. J. Hubschman; 2135572 of Nov. 8, 1938 to W. B. Fried; 2139620 of Dec. 6, 1938 to Lester Kirschbraun; 2151220 of Mar. 21, 1939 to L. H. Mattes; 2163757 of Jun. 27, 1939 to R. S. Maclean and C. W. Pollard; 2164725 of Jul. 4, 1939 to Gilbert Snyder; 2205798 of Jun. 25, 1940 to L. H. Mattes; 2207689 of Jul. 9, 1940 to G. J. Snyder; 2210321 of Aug. 6, 1940 to E. C. Knoblock; 2214387 of Sep. 10, 1940 to G. J. Snyder;

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2219723 of Oct. 29, 1940 to H. A. Mulderink and Mark Drinkall; 2223305 of Nov. 26, 1940 to S. F. Manning; 2223628 of Dec. 3, 1940 to Arthur Landis; 2224351 of Dec. 10, 1940 to R. L. Kaye; 2233854 of Mar. 4, 1941 to G. J. Snyder; 2235652 of Mar. 18, 1941 to G. J. Snyder; 2238787 of Apr. 15, 1941 to N. L. Aberson; 2243056 of May 20, 1941 to G. J. Snyder and R. N. Geoffroy; 2246377 of Jun. 17, 1941 to L. H. Mattes; 2249027 and 2249028 of Jul. 15, 1941 to H. A. Mulderink; 2252539 of Aug. 12, 1941 to F. W. Adams; 2255736 of Sep. 9, 1941 to L. S. Odell; 2268278 of Dec. 30, 1941 to Abbott Coburn; Design 131018 of Jan. 6, 1942 to Abbott Coburn; 2270808 and 2270809 of Jan. 20, 1942 to R. L. Kaye; Design 131154 of Jan. 20, 1942 to R. L. Kaye; 2278289 of Mar. 31, 1942 to G. J. Snyder; 2281724 and 2281725 of May 5, 1942 to G. J. Snyder; 2282432 of May 12, 1942 to G. J. Snyder; Design 133920 of Sep. 29, 1942 to Clayton Beeson; Design 133925 of Sep. 29, 1942 to G. W. Strunk and George Lehr; 2305280 of Dec. 15, 1942 to G. W. Strunk and George Lehr; 2308589 of Jan. 19, 1943 to W. G. Davis; Designs 135475, 135476 and 135477 of Apr. 13, 1943 to W. R. Wright; 2321396 and 2321397 of Jun. 8, 1943 to H. C. Koch; 2323299 of Jul. 6, 1943 to E. L. Craig; 2335493 of Nov. 30, 1943 to Mark Drinkall; 2339489 of Jan. 18, 1944 to Joseph Kublanow; Reissue 22481 of May 16, 1944 to R. L. Kaye; *Can. Pats.* 369467 of Oct. 26, 1937 to Building Products, Ltd.; 377957 of Nov. 29, 1938 to Mastic Asphalt Corp.; 383106 of Aug. 1, 1939 to Mastic Asphalt Corp.; 385832 of Dec. 26, 1939 to G. J. Snyder; 388792 of May 21, 1940 to Briktex Inc.; 392344 of Nov. 5, 1940 to Patent & Licensing Corp.; 392346 of Nov. 5, 1940 to Patent & Licensing Corp.; 395208 of Mar. 11, 1941 to H. C. Koch (Abbott Coburn); 397130 of Jun. 10, 1941 to Mastic Asphalt Corp.; 398880, 398881 and 398882 of Aug. 26, 1941 to Mastic Asphalt Corp.; 399547 of Sep. 23, 1941 to Building Products, Ltd.; 405316 of Jun. 9, 1942 to Mastic Asphalt Corp.; 412689 of May 25, 1943 to Homer Dufresne; 414786 of Aug. 31, 1943 to Abbott Coburn; 414864 of Aug. 31, 1943 to Building Products, Ltd. [See also Reference p. 771 (527).]

p. 793 (690) U. S. Pat. 2316345 of Apr. 13, 1943 to John Logan, Jr.

p. 793 (691) U. S. Pats. 1551662 of Sep. 1, 1925 to W. T. Hofmann; 1674630 of Jun. 26, 1928 to B. C. Beckman; 1924650 of Aug. 29, 1933 to G. B. Payne; 2037507 of Apr. 14, 1936 to A. C. Fischer; *Can. Pats.* 252359 of Aug. 4, 1925 to Beckman Dawson Roofing Co.; 379516 of Feb. 7, 1939 to W. B. Robinson.

p. 793 (692) U. S. Pat. 1438780 of Dec. 12, 1922 to Joseph O'Neil; 1702609 of Feb. 19, 1929 to E. W. Leshner; 2013218 of Sep. 3, 1935 to Joseph O'Neil.

p. 794 (693) U. S. Pat. 1473253 of Nov. 6, 1923 to R. V. Reynolds.

p. 794 (694) U. S. Pats. 1431125 and 1431126 of Oct. 3, 1922 to C. G. Robinson; 1926257 of Sep. 12, 1933 to R. B. Bawtenheimer; 2018216 of Oct. 22, 1935 to R. S. Maclean; 2021577, 2021578 and 2021579 of Nov. 19, 1935 to L. S. Odell; 2063935 of Dec. 15, 1936 to Lester Kirschbraun; 2085764 of Jul. 6, 1937 to L. S. Odell; 2131477 of Sep. 27, 1938 to Lester Kirschbraun; 2156566 of May 2, 1939 to Lester Kirschbraun; 2228362 of Jan. 14, 1941 to Robert Patterson; 2241898 of May 13, 1941 to R. B. Bawtenheimer; 2245047 of Jun. 10, 1941 to L. S. Odell; 2246660 of Jun. 24, 1941 to R. B. Bawtenheimer; 2266334 of Oct. 28, 1941 to S. S. Phillips; *Can. Pats.* 325428 of Aug. 30, 1932 to N. L. Aberson; 332475 of May 16, 1933 to Building Products, Ltd.; 336442 of Oct. 17, 1933 to Building Products, Ltd.; 339347 of Feb. 13, 1934 to Building Products, Ltd.; 347671 of Jan. 22, 1935 to Building Products, Ltd.; 351333 and 351334 of Jul. 2, 1935 to Building Products, Ltd.; 372370 of Mar. 8, 1938 to Charles Weir; 374826 of Jul. 5, 1938 to Building Products, Ltd.; 379090 of Jan. 24, 1939 to Building Products, Ltd.; 386872 of Feb. 13, 1940 to Patent & Licensing Corp.; 390763 and 390767 of Aug. 13, 1940 to Charles Weir; 403597 of Mar. 24, 1942 to Building Products, Ltd.; 404213 of Apr. 21, 1942 to Building Products, Ltd.

p. 794 (695) U. S. Pat. 2032083 of Feb. 25, 1936 to W. G. Dudleston.

p. 794 (696) U. S. Pats. 2006270 of Jun. 25, 1935 to N. P. Harshberger; 2201175 of May 21, 1940 to N. P. Harshberger.

p. 794 (697) U. S. Pat. 2209271 of Jul. 23, 1940 to N. P. Harshberger.

p. 794 (698) U. S. Pats. 2231006 of Feb. 11, 1941 to N. P. Harshberger; 2231007 of Feb. 11, 1941 to A. S. Vane; 2231008 of Feb. 11, 1941 to S. A. Ochs; *Can. Pats.* 395107, 395108 and 395109 of Mar. 11, 1941 to Bakelite Building Products Co., Inc.

- p. 794 (699) U. S. Pat. 2116452 of May 3, 1938 to G. E. Shipway.
- p. 794 (700) U. S. Pat. 1840244 of Jan. 5, 1932 to J. J. Molnar.
- p. 794 (701) U. S. Pats. 1886363 of Nov. 8, 1932 to F. C. Aufderheide; 1889091 of Nov. 29, 1932 to W. B. Fried; 2230702 of Feb. 4, 1941 to August Siebert.
- p. 794 (702) U. S. Pat. 2293331 of Aug. 18, 1942 to B. G. Dahlberg.
- p. 794 (703) U. S. Pat. 2298983 of Oct. 13, 1942 to E. R. Stabe.
- p. 795 (704) U. S. Pats. 1926093 of Sep. 12, 1933 to S. S. Gibney; 2018805 of Oct. 29, 1935 to Antoinette Reising; 2039536 of May 5, 1936 to W. D. Johnson; 2114451 of Apr. 19, 1938 to L. H. Mattes; Can. Pat. 372877 of Mar. 29, 1938 to F. A. Taschereau.
- p. 795 (705) U. S. Pat. 742589 of Oct. 27, 1903 to B. G. Casler.
- p. 795 (706) U. S. Pat. 816522 of Mar. 27, 1906 to G. R. Wyman.
- p. 795 (707) U. S. Pat. 1119553 of Dec. 1, 1914 to T. M. Vaughan; Can. Pat. 361274 of Oct. 20, 1936 to Crescent Brass & Pin Co.
- p. 795 (708) "Standard Method of Test for Weight of Coating on Zinc-Coated (Galvanized) Iron or Steel Articles" (A 90-39), A.S.T.M. Standards 1942, I, 534; "Recommended Practice for Safeguarding Against Embrittlement of Hot Galvanized Structural Steel Products and Procedure for Detecting Embrittlement" (A 143-35), A.S.T.M. Standards 1942, I, 543; "Tentative Specifications for Electrodeposited Coatings of Zinc on Steel" (A 164-40T), A.S.T.M. Standards 1942, I, 1448; "Tentative Specifications for Electrodeposited Coatings of Cadmium on Steel" (A 165-40T), A.S.T.M. Standards 1942, I, 1451; "Tentative Methods of Test for Local Thickness of Electrodeposited Coatings" (A 219-40T), A.S.T.M. Standards 1942, I, 1465; "Standard Method of Test for Uniformity of Coating by the Preece Test (Copper Sulfate Dip) on Zinc-Coated (Galvanized) Iron or Steel Articles" (A 239-41), A.S.T.M. Standards 1942, I, 538; American Assoc. State Highway Officials Standards A.A.S.H.O.: T 65-42 and T 66-42.
- p. 795 (709) U. S. Pats. 795553 of Jul. 25, 1905 to H. B. Sherman; 1435134 of Nov. 7, 1922 to Ernest Boley; 2111110 of Mar. 15, 1938 to A. J. Deniston, Jr. and E. D. Tripp; 2207897 of Jul. 16, 1940 to W. C. Schaus; 2334406 of Nov. 16, 1943 to A. J. Gray; Ger. Pat. Design 280101 of May 14, 1906 to Fritz Schmidt.
- p. 795 (710) U. S. Pats. 1410076 of Mar. 21, 1922 to F. C. Overbury; 1940534 of Dec. 19, 1933 to Edward Caughey; 2139279 of Dec. 6, 1938 to H. M. Maze; Can. Pat. 384737 of Oct. 17, 1939 to A. J. Deniston, Jr. and E. D. Tripp.
- p. 795 (711) U. S. Pat. 2001932 of May 21, 1935 to Hamilton Maze; Can. Pats. 347620 of Jan. 22, 1935 to A. J. Deniston, Jr.; 402378 of Jan. 20, 1942 to W. H. Maze Co.
- p. 795 (712) U. S. Pats. 757193 of Apr. 12, 1904 to F. S. Howard; 778863 of Jan. 3, 1905 to F. S. Howard; 1978190 of Oct. 23, 1934 to C. C. Figge; 2057003 of Oct. 13, 1936 to Clarence Bugher; Ger. Pats. 178525 of Jun. 22, 1905 to C. J. Carroll and J. H. Purnell; Design 387381 of Jul. 24, 1909 to Ludwig Kerkow Dachpappenfabrik.
- p. 796 (713) U. S. Pats. 954995 of Apr. 12, 1910 to W. E. Polhemus; 1524090 of Jan. 27, 1925 to Dozier Finley; 1595079 of Aug. 10, 1926 to A. C. Fischer; 1881438 of Oct. 11, 1932 to A. C. Fischer; Ger. Pat. Appl. C-49370 of Jun. 29, 1934 to Aktieselskabet Jens Villadsens Fabriker.
- p. 796 (714) Ger. Pat. Design 1439718 of Nov. 30, 1937 to Alex Rump.
- p. 796 (715) U. S. Pats. 1592760 of Jul. 13, 1926 to A. C. Fischer; 1743764 of Jan. 14, 1930 to A. C. Fischer; 1769628 of Jul. 1, 1930 to A. C. Fischer; 1848076 of Mar. 1, 1932 to A. C. Fischer; 1849869 of Mar. 15, 1932 to A. C. Fischer; 1905376 of Apr. 25, 1933 to A. C. Fischer; 1983494 and 1983495 of Dec. 4, 1934 to A. C. Fischer; Can. Pat. 397918 of Jul. 15, 1941 to L. A. Hollister.
- p. 796 (716) U. S. Pats. 1241146 of Sep. 25, 1917 to R. P. Perry; 1805739 of May 19, 1931 to W. J. Moeller and A. H. Zimmerman; 2222868 of Nov. 26, 1940 to L. A. Hollister; Russian Pat. 53780 of Aug. 31, 1938 to N. V. Mikhailov and P. E. Gukalov.
- p. 796 (717) "Underwriters' Laboratories Standards for Class C, Asphalt Ragfelt Sheet Roofing and Shingles," issued by the Underwriters' Laboratories, Inc., Chicago, Ill., Edition of Jan. 2, 1941; "Federal Spec. for Roofing; Asphalt-Prepared, Smooth-Surfaced," SS-R-501, Federal Standard Stock Catalog, Section IV (Part 5), Aug. 1, 1933; "Federal Spec. for Roof-

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ing and Shingles; Asphalt-Prepared, Mineral-Surfaced," SS-R-521, Federal Standard Stock Catalog, Section IV (Part 5), Aug. 1, 1933; "Specifications for Asphalt Roofing Surfaced with Powdered Talc or Mica" (D 224-41T), A.S.T.M. Standards, 1942, II, 1311; "Tentative Specs. for Asphalt Roofing Surfaced with Coarse Mineral Granules" (D 249-42T), A.S.T.M. Standards, 1942, II, 1307.

p. 796 (718) U. S. Pat. 1481508 of Jan. 22, 1924 to C. A. Harris.

p. 796 (719) U. S. Pats. 1602314 of Oct. 5, 1926 to A. J. Signor; Reissue 18449 of May 3, 1932 to J. A. Signor; 2008952 of Jul. 23, 1935 to Erich Gach.

p. 797 (720) U. S. Pat. 1968281 of Jul. 31, 1934 to D. C. Cale.

p. 797 (721) U. S. Pat. 1925961 of Sep. 5, 1933 to N. P. Harshberger.

p. 797 (722) U. S. Pats. 134702 of Jan. 7, 1873 to R. U. Piper; 348844 of Sep. 7, 1886 to David Harger; 1109738 of Sep. 8, 1914 to W. P. Coldren; 1263218 and 1263219 of Apr. 16, 1918 to A. C. Fischer; 1332223 of Mar. 2, 1920 to J. R. McCord, Jr.; 1334178 of Mar. 16, 1920 to W. B. Sharp; 1446455 of Feb. 27, 1923 to A. C. Fischer; 1447986 of Mar. 13, 1923 to W. C. Johnson; 1480246 of Jan. 8, 1924 to C. E. Douglas; 1600667 of Sep. 21, 1926 to A. C. Fischer; 1626802 of May 3, 1927 to A. C. Fischer; Ger. Pats. 188146 of Feb. 4, 1905 to Roland Risse; 286903 of Sep. 20, 1913 to Ludwig Schwabe; Design 82696 of Aug. 25, 1897 to Karl Voss.

p. 797 (723) U. S. Pats. 255087 of Mar. 14, 1882 to L. L. Sagendorph; 257616 of May 9, 1882 to F. E. Sagendorph; 259228 of Jun. 6, 1882 to O. A. Smith and F. L. Kane; 291440 of Jan. 1, 1884 to C. M. Warren; 362246 of May 3, 1887 to J. D. Brown, Jr.; 362732 of May 10, 1887 to W. H. Fay; 754273 of Mar. 8, 1904 to W. H. Bache; 876843 of Jan. 14, 1908 to A. E. Roever; 936139 of Oct. 5, 1909 to Charles Lagergren; 954995 of Apr. 12, 1910 to W. E. Polhemus; 984860 of Feb. 21, 1911 to F. E. Smith; 1087321 of Feb. 17, 1914 to Calvin Russell; 1276632 of Aug. 20, 1918 to Ernest Flagg; 1356930 of Oct. 26, 1920 to J. R. McCord, Jr.; 1471156 of Oct. 16, 1923 to A. A. Griswold; 1612718 of Dec. 28, 1926 to J. F. Grice; 1618902 of Feb. 22, 1927 to Henry Yost; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1684179 of Sep. 11, 1928 to A. A. Griswold; 1747492 of Feb. 18, 1930 to J. S. Skoglund and C. M. Engebretson; 1755538 of Apr. 22, 1930 to A. H. Draughon, Jr.; 1794072 of Feb. 24, 1931 to A. A. Griswold; 1810808 of Jun. 16, 1931 to Henry Yost; 1825732 of Oct. 6, 1931 to G. A. Holman; 1952383 of Mar. 27, 1934 to F. C. McClanahan; 1978237 of Oct. 23, 1934 to O. P. Valiquet and J. F. Chandler; 1990776 of Feb. 12, 1935 to C. J. Dexter; 2060739 of Nov. 10, 1936 to Homer Maddux; Reissue 20470 of Aug. 17, 1937 to C. J. Dexter; 2117014 of May 10, 1938 to E. L. Black; 2176344 of Oct. 17, 1939 to J. B. Hunt; 2205520 of Jun. 25, 1940 to Joseph Farkas; Can. Pats. 234217 of Sep. 18, 1923 to H. P. Baltzer; 278134 of Feb. 28, 1928 to Henry Yost; 329550 of Jan. 24, 1933 to G. A. Holman; 351115 of Jun. 25, 1935 to O. P. Valiquet; Ger. Pats. 188146 of Feb. 4, 1905 to Roland Risse; 189198 of Sep. 19, 1905 to Julius Fichtel; Design 387381 of Jul. 24, 1909 to Ludwig Kerkow Dachpappenfabrik; Design 1269843 of Jul. 7, 1933 to Hans Meier.

p. 797 (724) U. S. Pats. 887532 of May 12, 1908 to H. B. Sherman; 967208 of Aug. 16, 1910 to J. F. Leslie; 1321958 of Nov. 18, 1919 to H. R. Wardell; 2166064 of Jul. 11, 1939 to Waldemar Kroier; Can. Pat. 139036 of Mar. 12, 1912 to H. W. Johns-Manville Co.

p. 797 (725) U. S. Pats. 973902 of Oct. 25, 1910 to W. H. Woerheide; 981362 of Jan. 10, 1911 to J. H. Bell; 985501 of Feb. 28, 1911 to J. H. Bell; 1017611 of Feb. 13, 1912 to H. R. Wardell; 1018946 of Feb. 27, 1912 to H. W. Topping; 1055848 of Mar. 11, 1913 to W. H. Woerheide; 1066959 of Jul. 8, 1913 to J. A. Topping; 1076639 of Oct. 21, 1913 to W. H. Nicholls and A. C. Carmichael; 1101896 of Jun. 30, 1914 to W. H. Woerheide; 1109738 of Sep. 8, 1914 to W. P. Coldress; 1148260 of Jul. 27, 1915 to P. W. Stansbury; 1148647 of Aug. 3, 1915 to E. J. Yetter; 1150261 of Aug. 17, 1915 to A. C. Fischer; 1163269 of Dec. 7, 1915 to H. B. Sherman; 1186257 of Jun. 6, 1916 to W. H. Woerheide; 1187532 of Jun. 20, 1916 to H. C. Kettelson; 1273500 of Jul. 23, 1918 to H. C. Kettelson; 1348858 of Aug. 10, 1920 to L. M. Ford; 1357265 of Nov. 2, 1920 to W. H. Woerheide; 1642088 of Sep. 13, 1927 to T. M. Scarff; 1657851 of Jan. 31, 1928 to D. R. Bard; 1692401 of Nov. 20, 1928 to D. R. Bard; 1761599 of Jun. 3, 1930 to J. A. Topping; 1930632 of Oct. 17, 1933 to J. A. Topping; Can.

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Pats. 284462 of Nov. 6, 1928 to D. R. Bard; 335689 of Sep. 12, 1933 to J. A. Topping; 354586 of Dec. 3, 1935 to Galt Art Metal Co.; **Brit. Pat.** 162043 of Apr. 18, 1921 to Evelyn Hurden, A. P. Hurden and S. E. Beeson; **Ger. Pats.** 139645 of Jun. 8, 1902 to Anton Köstner; 188146 of Feb. 4, 1905 to Roland Risse; **Design** 402505 of Nov. 2, 1909 to Willibald Eulha; **Design** 474583 of Jul. 10, 1911 to Fritz Sonnemann; 275332 of Aug. 19, 1911 to J. T. Neuhaus.

p. 797 (726) **U. S. Pats.** 757193 of Apr. 12, 1904 to F. S. Howard; 778863 of Jan. 3, 1905 to F. S. Howard; 1052258 of Feb. 4, 1913 to E. J. Kenney; 1225972 of May 15, 1917 to H. C. Kettelson; 1237270 of Aug. 21, 1917 to Herbert Abraham; 1242675 of Oct. 9, 1917 to S. M. Ford; 1297323 of Mar. 18, 1919 to R. C. Clark; 14711156 of Oct. 16, 1923 to A. A. Griswold; 1479067 of Jan. 1, 1924 to A. A. Griswold; **Can. Pat.** 97905 of Mar. 6, 1906 to F. S. Howard; **Brit. Pat.** 487155 of Jun. 13, 1938 to B. C. Kridler.

p. 797 (727) **U. S. Pats.** 60708 of Jan. 1, 1867 to C. J. Fay; 61184 of Jan. 15, 1867 to L. D. Ford; 154843 of Sep. 8, 1874 to Rowell Colby; 223671 of Jan. 20, 1880 to Thomas Fugate; 257616 of May 9, 1882 to F. E. Sagendorph; 259228 of Jun. 6, 1882 to O. A. Smith and F. L. Kane; 285131 of Sep. 18, 1883 to J. F. Hoffman; 322153 of Jul. 14, 1885 to L. F. Blair and J. W. Roche; 332570 of Dec. 15, 1885 to W. H. Stewart; 352619 of Nov. 16, 1886 to F. L. Kane; 354311 of Dec. 14, 1886 to C. A. Favel; 362246 of Mar. 3, 1887 to J. D. Brown, Jr.; 362732 of May 10, 1887 to W. H. Fay; 632825 of Sep. 12, 1899 to R. J. Redick; 636022 of Oct. 31, 1899 to G. D. Crabbs and W. H. Pendery; 652150 of Jun. 19, 1900 to F. W. Terpenning; 669315 of Mar. 5, 1901 to W. P. Whitmore; 713588 of Nov. 18, 1902 to John Ayrault, Jr.; 754273 of Mar. 8, 1904 to W. H. Bache; 762220 of Jun. 7, 1904 to Jordan Williams (metal); 813163 of Feb. 20, 1906 to W. J. Moeller; 821606 of May 29, 1906 to E. N. Brogan; 835899 of Nov. 13, 1906 to W. J. Moeller; 855757 of Jun. 4, 1907 to G. D. Crabbs and W. H. Pendery; 868930 of Oct. 22, 1907 to A. E. Kirk; 876843 of Jan. 14, 1908 to A. E. Roeveer; 936139 of Oct. 5, 1909 to Charles Lagergren; 948116 of Feb. 1, 1910 to W. H. Pendery; 972957 of Oct. 18, 1910 to H. R. Wardell; 984860 of Feb. 21, 1911 to F. E. Smith; 1018524 of Feb. 27, 1912 to J. J. Smiley; 1083243 of Dec. 30, 1913 to W. C. Edwards, Jr.; 1087321 of Feb. 17, 1914 to Calvin Russell; 1100955 of Jun. 23, 1914 to E. B. Coburn; 1158266 of Oct. 26, 1915 to F. C. Overbury and H. C. Platts; 1338262 of Apr. 27, 1920 to C. A. Statler; 1362755 of Dec. 21, 1920 to C. A. Statler; 1372208 of Mar. 22, 1921 to C. A. Statler; 1434200 of Oct. 31, 1922 to W. M. Chase; 1435623 of Nov. 14, 1922 to I. C. Saxe; 1447986 of Mar. 13, 1923 to W. C. Johnson; 1489898 of Apr. 8, 1924 to Svend Petersen; 1552883 of Sep. 8, 1925 to Richard Rudel; 1568227 of Jan. 5, 1926 to F. P. Leonard; 1600667 of Sep. 1, 1926 to A. C. Fischer; 1655494 of Jan. 10, 1928 to C. P. Cowan; 1684179 of Sep. 11, 1928 to A. A. Griswold; 1690792 of Nov. 6, 1928 to R. C. Neptune; 1710104 of Apr. 23, 1929 to R. C. Neptune; 1723927 of Aug. 6, 1929 to A. C. Fischer; 1829488 of Oct. 27, 1931 to E. C. Miller; 1873944 of Aug. 23, 1932 to J. E. Black; 1882177 of Oct. 11, 1932 to L. W. Burris; 1950840 of Mar. 13, 1934 to J. E. Cook; 2003503 of Jun. 4, 1935 to S. L. Eason; 2003728 of Jun. 4, 1935 to Hermann von Forster and Samuel de Lange; 2013330 of Sep. 3, 1935 to L. M. Abraham; 2125694 of Aug. 2, 1938 to Phillip Sattig; 2153887 of Apr. 11, 1939 to H. W. Greider and G. A. Fasold; 2160845 of Jun. 6, 1939 to S. L. Eason; 2215349 of Sep. 17, 1940 to S. L. Eason; 2241058 of May 6, 1941 to S. L. Eason; 2305008 of Dec. 15, 1942 to Orlie Howard; **Can. Pats.** 29795 of Sep. 1, 1885 to W. H. Fay; 84226 of Dec. 8, 1903 to John Ayrault; 98890 and 98891 of May 8, 1906 to Philip Carey Mfg. Co.; 107146 of Aug. 27, 1907 to E. N. Brogan; 117009 of Mar. 2, 1909 to A. E. Kirk; 145536 of Jan. 28, 1913 to Calvin Russell; 218121 of May 2, 1922 to Fred Frederiksen; 247225 of Mar. 3, 1925 to Svend Petersen; 259005 of Mar. 16, 1926 to Bird & Son, Ltd.; 260351 of May 4, 1926 to J. A. Blair; 364370 of Feb. 23, 1937 to S. L. Eason; 372991 of Apr. 5, 1938 to R. S. Chappell; 408927 of Dec. 1, 1942 to S. L. Eason; **Brit. Pat.** 460550 of Apr. 20, 1936 to Phillip Sattig; **Ger. Pats.** 53010 of Dec. 29, 1889 to Adalbert Kelm; 72880 of Apr. 23, 1893 to E. Fischer; **Design** 82696 of Aug. 25, 1897 to Karl Voss; 119104 of May 2, 1900 to Paul Fuhrmann; 574880 of Aug. 31, 1929 to Wacław Szpakowski; 638035 of Jun. 30, 1934 to Aktielselskabet Jens Villardsens Fabrikker; **Appl.** W-95428 of Nov. 17, 1934 to Fritz Werner; 647385 of Nov. 18, 1934 to Fritz Werner; **Design** 1415632 of Feb.

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11, 1936 to Fritz Werner; Appl. S-118059 of Apr. 20, 1935 to Phillip Sattig; *Austrian Pat.* 65252 of Dec. 15, 1913 to Heinrich Schwarz.

p. 797 (728) *U. S. Pats.* 61899 of Feb. 5, 1867 to I. C. Wands; 97796 of Dec. 14, 1869 to H. G. Noble; 143471 of Oct. 7, 1873 to R. A. Smith; 195593 of Sep. 25, 1877 to H. F. Evans; 511385, 511386 and 511387 of Dec. 26, 1893 to James Smith; 674579 of May 21, 1901 to W. H. Grow; 993686 of May 30, 1911 to F. S. Howard; 1051702 of Jan. 28, 1913 to H. W. Deming; 1061685 of May 13, 1913 to George Newton; 1137895 of May 4, 1915 to A. W. Phippen; 1144265 of Jun. 22, 1915 to Herman von Uffel; 1147582 of Jul. 20, 1915 to Herman von Uffel; 1158267 and 1158268 of Oct. 26, 1915 to F. C. Overbury and H. C. Platts; 1210855 of Jan. 2, 1917 to R. L. Shainwald, Jr.; 1221370 of Apr. 3, 1917 to F. C. Overbury and H. C. Platts; 1247743 of Nov. 27, 1917 to W. D. Thompson; 1421040 of Jun. 27, 1922 to G. E. Swenson; 1447175 of Mar. 6, 1923 to O. L. Henderson; 1517944 of Dec. 2, 1924 to J. F. Bobbitt; 1544866 of Jul. 7, 1925 to G. F. Slater; 1808286 of Jun. 2, 1931 to G. G. Britton; 1908127 of May 9, 1933 to W. T. Deacon; 2110402 of Mar. 8, 1938 to W. H. Robison; 2220259 of Nov. 5, 1940 to T. C. McPherson; *Ger. Pats.* 24612 of Oct. 5, 1882 to David Röhm; 44463 of Dec. 24, 1887 to Julius Wascher; 51585 of Jun. 5, 1889 to Ludwig Hatschek; Design 84880 of Nov. 8, 1897 to Karl Schultheiss; Design 402505 of Nov. 2, 1909 to Willibald Elsel; 275332 of Aug. 19, 1911 to J. T. Neuhaus; 292077 of Jan. 20, 1915 to Hans Christen; Design 783579 of Mar. 26, 1920 to Otto Faust; 574880 of Aug. 31, 1929 to Wacław Szpakowski.

p. 797 (729) *U. S. Pats.* 1986471 of Jan. 1, 1935 to A. C. Fischer; 2133683 of Oct. 18, 1938 to E. R. Black.

p. 797 (730) *U. S. Pats.* 1901356 of Mar. 14, 1933 to J. H. Plunkett; 2023030 of Dec. 3, 1935 to J. H. Plunkett; *Can. Pats.* 342629 of Jun. 26, 1934 to Patent & Licensing Corp.; 362410 of Dec. 8, 1936 to Patent & Licensing Corp.

p. 797 (731) *U. S. Pat.* 2123401 of Jul. 12, 1938 to R. R. Clements.

p. 797 (732) *U. S. Pat.* 1008435 of Nov. 14, 1911 to H. M. Reynolds.

p. 797 (733) *U. S. Pat.* 694304 of Feb. 25, 1902 to C. S. Bird and J. B. Hanscom.

p. 797 (734) *Can. Pat.* 354392 of Nov. 26, 1935 to Building Products, Ltd.

p. 797 (735) *U. S. Pat.* 803713 of Nov. 7, 1905 to H. M. Reynolds.

p. 797 (736) *U. S. Pat.* 852397 of Apr. 30, 1907 to W. L. Penney.

p. 797 (737) *U. S. Pat.* 713938 of Nov. 18, 1902 to W. H. Bache.

p. 797 (738) *U. S. Pat.* 873046 of Dec. 10, 1907 to F. S. Howard.

p. 797 (739) *U. S. Pats.* 742558 of Oct. 27, 1903 to W. H. Bache; 874160 of Dec. 17, 1907 to Purlan Buckborough.

p. 797 (740) *U. S. Pats.* 825239 of Jul. 3, 1906 to M. C. Ohnemus; 919607, 919608 and 919739 of Apr. 27, 1909 to G. W. Loughman; 923362 of Jun. 1, 1909 to G. W. Loughman; 1029947 of Jun. 18, 1912 to C. J. Rothermel; 1517940 of Dec. 2, 1924 to J. W. Bellairs; 1548632 of Aug. 4, 1925 to C. J. Rothermel; 1912388 of Jun. 6, 1933 to Samuel Slovin; *Can. Pat.* 99185 of May 22, 1906 to M. C. Ohnemus.

p. 797 (741) *U. S. Pats.* 996510 and 996511 of Jun. 27, 1911 to G. J. Oltsch; 1298811 of Apr. 1, 1919 to A. S. Spiegel; 1781016 of Nov. 11, 1930 to Henry Kielberg; *Can. Pat.* 207018 of Dec. 28, 1920 to Roofing Patents Co.

p. 797 (742) *U. S. Pats.* 1312202 of Aug. 5, 1919 to R. P. Perry; 1312211 of Aug. 5, 1919 to C. J. Rothermel.

p. 798 (743) *U. S. Pats.* 980406 of Jan. 3, 1911 to B. G. Casler; 1543052 of Jun. 23, 1925 to A. W. Brown.

p. 798 (744) *U. S. Pats.* 1651664 of Dec. 6, 1927 to C. E. Bowers; 2146704 of Feb. 14, 1939 to E. M. E. Anderson.

p. 798 (745) *U. S. Pat.* 1572140 of Feb. 9, 1926 to O. A. Heppes; *Can. Pat.* 143374 of Oct. 15, 1912 to C. W. Dohm.

p. 798 (746) *U. S. Pat.* 1380475 of Jun. 7, 1921 to O. A. Heppes.

p. 798 (747) *U. S. Pat.* 1365403 of Jan. 11, 1921 to R. F. Holway.

p. 798 (748) *U. S. Pats.* 1312202 of Aug. 5, 1919 to R. P. Perry; 1312211 of Aug. 5, 1919 to C. J. Rothermel.

- p. 798 (749) U. S. Pats. 1359569 of Nov. 23, 1920 to C. S. Bird; 1429836 of Sep. 19, 1922 to C. S. Bird; 1765839 of Jun. 24, 1930 to Lester Kirschbraun; Design 102555 of Dec. 29, 1936 to H. W. Holland.
- p. 798 (750) U. S. Pat. 1873887 of Aug. 23, 1932 to O. A. Heppes.
- p. 798 (751) U. S. Pats. 1324039 of Dec. 9, 1919 to C. F. Evans; 1460833 of Jul. 3, 1923 to Herbert Abraham; 1482764 of Feb. 5, 1924 to George Ritter; 1493616 of May 13, 1924 to J. E. Domagall; 1686513 of Oct. 9, 1928 to W. H. Cady.
- p. 798 (752) U. S. Pats. 1604182 and 1604183 of Oct. 26, 1926 to J. P. Markert.
- p. 798 (753) Can. Pat. 140335 of May 14, 1912 to E. H. Cunningham.
- p. 798 (754) Ger. Pat. Design 1250165 of Jan. 9, 1933 to Nikolaus Jungblut.
- p. 798 (755) U. S. Pat. 840103 of Jan. 1, 1907 to R. W. Bird; Ger. Pat. Appl. Sch-111.30 of Dec. 24, 1930 to Hans Schmitt.
- p. 798 (756) U. S. Pats. 372316 of Nov. 1, 1887 to C. B. Hutchins; 761138 of May 31, 1904 to C. S. Bird and P. R. Allen; 845414 of Feb. 26, 1907 to Samuel Herbert.
- p. 799 (757) U. S. Pat. 1928751 of Oct. 3, 1933 to T. S. Bradley.
- p. 799 (758) U. S. Pat. 1166541 of Jan. 4, 1916 to F. C. Overbury.
- p. 799 (759) Can. Pat. 254431 of Oct. 6, 1925 to Paraffine Cos., Inc.
- p. 799 (760) U. S. Pats. 1418377 of Jun. 6, 1922 to C. B. Jaynes; 1467017 of Sep. 4, 1923 to G. C. Trice.
- p. 799 (761) U. S. Pat. 2203312 of Jun. 4, 1940 to G. E. Swenson.
- p. 799 (762) U. S. Pats. 18186 of Sep. 15, 1857 to W. H. Carver and J. Beckley; 40542 of Nov. 3, 1863 to L. S. Mills and C. H. Smith; 55891 of Jun. 26, 1866 to Rufus Norwood; 58246 of Sep. 25, 1866 to William Gilbert; 61787 of Feb. 5, 1867 to J. R. Woodworth; 61878 of Feb. 5, 1867 to John Scanlan; 63429 of Apr. 2, 1867 to F. O. Rogers; 64493 of May 7, 1867 to Marvin Converse and A. C. Torry; 74430 of Feb. 11, 1868 to Leander Rodney; 76375 of Apr. 7, 1868 to Edward Atkinson; 82507 of Sep. 29, 1868 to T. E. Wood; 93304 of Aug. 3, 1869 to C. B. Hutchins; 94493 of Sep. 7, 1869 to Benjamin Hinkley; 101071 of Mar. 22, 1870 to T. E. Wood; 102061 of Apr. 19, 1870 to W. M. Stuart; 124192 of Mar. 5, 1872 to D. W. Bailey; 136516 of Mar. 4, 1873 to J. H. Hood; 140945 of Jul. 15, 1873 to Charles Mueller; 146608 of Jan. 20, 1874 to Tobias New; 147962 of Feb. 24, 1874 to Tobias New; 156537 of Nov. 3, 1874 to Edward Churchill; 156639 of Nov. 10, 1874 to Jeduthan Kittredge; 161762 of Apr. 6, 1875 to Allan Cummings; 179131 of Jun. 27, 1876 to Lewis Peirce; 197170 of Nov. 13, 1877 to Lewis Peirce; 199210 of Jan. 15, 1878 to F. L. Kane; 200122 of Feb. 12, 1878 to M. B. Bailey; 202493 of Apr. 16, 1878 to E. B. Warren; 205132 of Jun. 18, 1878 to Lewis Peirce; Reissue 8414 of Sep. 10, 1878 to Tobias New; 209705 of Nov. 5, 1878 to Tobias New; 209830 of Nov. 12, 1878 to Tobias New; 227682 of May 18, 1880 to S. L. Foster and W. H. Benton; Reissue 9314 of Jul. 20, 1880 to S. L. Foster and W. H. Benton; 236386 of Jan. 4, 1881 to C. M. Warren; 270943 of Jan. 23, 1883 to S. L. Foster; 296163 of Apr. 1, 1884 to Levi Haas and Dennis Howarth; 313971 of Mar. 17, 1885 to T. H. White; 438324 of Oct. 14, 1890 to M. W. Powell; 677058 of Jun. 25, 1901 to Emil Borgeson and Axel Wennerberg; 712193 of Oct. 28, 1902 to F. L. Kane; 720811 of Feb. 17, 1903 to John Ingram; 798131 of Aug. 29, 1905 to F. W. Gezelschap and Arthur Winding; 805745 and 805746 of Nov. 28, 1905 to F. N. Pease; 822602 of Jun. 5, 1906 to August Gross and A. C. Horn; 842079 of Jan. 22, 1907 to E. B. Campbell; 846572 of Mar. 12, 1907 to C. J. Kunzler; 879372 of Feb. 18, 1908 to C. C. Dill; 921538 of May 11, 1909 to John Glassford; 1012903 of Dec. 26, 1911 to Henry Olmsted, Jr.; 1044773 and 1044774 of Nov. 19, 1912 to Harry Gillett; 1110330 of Sep. 15, 1914 to F. J. McClaskey; 1170521 of Feb. 8, 1916 to S. B. Faison; 1207726 of Dec. 12, 1916 to C. V. Eades; 1230396 of Jun. 19, 1917 to F. L. Foster; 1235270 of Jul. 31, 1917 to J. B. Wise; 1261276 of Apr. 2, 1918 to J. C. Norton; 1284997 of Nov. 19, 1918 to O. A. Bigler; 1343478 of Jun. 15, 1920 to H. H. Robertson; 1363539 of Dec. 28, 1920 to J. O. Boylan; 1422526 of Jul. 11, 1922 to J. M. Berdan; 1606496 of Nov. 9, 1926 to C. J. Beckwith and R. K. Austin; 1705160 of Mar. 12, 1929 to J. P. Stagg; 1780622 of Nov. 4, 1930 to W. H. Lawrence; 1880429 of Oct. 4, 1932 to F. H. Ford; 1958871 of May 15, 1934 to E. M. Tucker; 1967105 of Jul. 17, 1934 to H. L. Seymour; 1994262 of Mar. 12, 1935 to Arthur Winding; 2007403 of Jul. 9, 1935 to H. C. Macan; 2144168 of Jan. 17, 1939 to Fred Sherriff; 2229535 of Jan. 21, 1941 to

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C. M. Weber and J. D. Long; *Can. Pats.* 7669 of Jul. 17, 1877 to Alexander McLean; 15035 of Jun. 28, 1882 to J. W. Paterson; 28896 of Apr. 15, 1888 to Clinton French; 216492 of Mar. 7, 1922 to Johns-Manville, Inc.; 278755 of Mar. 20, 1928 to Johns-Manville Corp.; 284700 of Nov. 13, 1928 to W. A. Moffatt; 347154 of Jan. 1, 1935 to Barrett Co.; 360554 of Sep. 15, 1936 to Anaconda Copper Mining Co.; *Brit. Pats.* of 1897 (Nov. 26), 27884 to Robert Glendenning; of 1898 (Dec. 23), 27118 to J. J. Ingham; 427851 of Jul. 13, 1934 to Jacques Gevers; 431351 of Jan. 5, 1934 to W. B. Ranson; 467425 of Jun. 16, 1937 to W. B. Ranson; 530518 of Dec. 13, 1940 to W. B. Ranson; *Ger. Pats.* 212527 of Oct. 30, 1906 to Ludwig Esselborn; Design 325524 of Sep. 11, 1907 to Fritz Grimm; Design 330871 of Feb. 28, 1907 to Wirtschaftliche Vereinigung für die Westdeutsche Dachpappenindustrie, G.m.b.H.; Design 336320 of Mar. 9, 1908 to B. Lohse & Rothe; Design 347273 of Mar. 20, 1907 to Büsscher & Hoffmann, G.m.b.H.; 219448 of Mar. 17, 1908 to Friedrich Drexler; Design 414575 of Sep. 3, 1909 to Albin Karg; Design 543093 of Apr. 16, 1912 to Ernst Zorn; Appl. G-108.30 of Jan. 16, 1928 to C. Gartenmann & Cie.; Appl. H-137428 of Sep. 9, 1933 to Heinrich Habig; Appl. D-70963 of Aug. 22, 1935 to Johann Drolshagen.

p. 799 (763) U. S. Pats. 61746 of Feb. 5, 1867 to R. O. Lowrey; 133247 of Nov. 19, 1872 to John Park; 147962 of Feb. 24, 1874 to Tobias New; Reissue 8414 of Sep. 10, 1878 to Tobias New; 209131 of Oct. 22, 1878 to Tobias New; 217916 and 217917 of Jul. 29, 1879 to C. M. Warren; 227682 of May 18, 1880 to S. L. Foster and W. H. Benton; Reissue 9314 of Jul. 20, 1880 to S. L. Foster and W. H. Benton; 318023 of May 19, 1885 to M. W. Powell; 712193 of Oct. 28, 1902 to F. L. Kane; 712308 of Oct. 28, 1902 to F. L. Kane; 2019647 of Nov. 5, 1935 to Frederick Basten and Evelyn Hurden; 2031249 of Feb. 18, 1936 to O. S. Bowman; *Brit. Pats.* of 1890 (Apr. 5), 2619 to Johann Schultz and Edward Hoff; 389668 of Apr. 28, 1932 to Laurent Noesen; 431351 of Jan. 5, 1934 to W. B. Ranson; 467425 of Dec. 16, 1935 to W. B. Ranson; *Ger. Pats.* 24612 of Oct. 5, 1882 to David Röhm; Design 419504 of Oct. 12, 1909 to Hilarius Knobel; Design 441025 of Mar. 14, 1910 to Hilarius Knobel.

p. 799 (764) "Federal Spec. for Roofing; Built-up, Type 5TWS (Construction of)," SS-R-578, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-179. (Withdrawn.)

p. 799 (765) "Federal Spec. for Roofing; Built-up, Type 5AWS (Construction of)," SS-R-572, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-171. (Withdrawn.)

p. 799 (766) "Federal Spec. for Roofing; Built-up, Type 5TCS (Construction of)," SS-R-575, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-177. (Withdrawn.)

p. 799 (767) "Federal Spec. for Roofing; Built-up, Type 5ACS (Construction of)," SS-R-569, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-174. (Withdrawn.)

p. 799 (768) "Federal Spec. for Roofing; Built-up, Type 4TWS (Construction of)," SS-R-566, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-178. (Withdrawn.)

p. 799 (769) "Federal Spec. for Roofing; Built-up, Type 4AWS (Construction of)," SS-R-560, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-170. (Withdrawn.)

p. 799 (770) "Federal Spec. for Roofing; Built-up, Type 4ACS (Construction of)," SS-R-557, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-173. (Withdrawn.)

p. 799 (771) "Federal Spec. for Roofing; Built-up, Type 4TCS (Construction of)," SS-R-563, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-176. (Withdrawn.)

p. 799 (772) "Federal Spec. for Roofing; Built-up, Type 3TCS (Construction of)," SS-R-554, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-175. (Withdrawn.)

p. 799 (773) "Federal Spec. for Roofing; Built-up, Type 3ACS (Construction of),"

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SS-R-551, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-172. (Withdrawn.)

p. 799 (774) "Federal Spec. for Flashings; Metal (Installation with Bituminous Built-up Roofing)," QQ-F-451, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-180. (Withdrawn.) "Federal Spec. for Flashings; Plastic (Installation with Bituminous Built-up Roofing)," SS-F-451, Federal Standard Stock Catalog, Section IV (Part 5); Bureau of Standards Circular C-181. (Withdrawn.)

p. 800 (775) U. S. Pats. 34653 of Mar. 11, 1862 to Zadok Street; 40542 of Nov. 3, 1863 to L. S. Miller; 94689 of Sep. 7, 1869 to R. K. Kille; Reissue 3701 of Nov. 2, 1869 to R. K. Kille; 141437 of Aug. 5, 1873 to A. G. Hennion; 145705 of Dec. 16, 1873 to Horace Wheeler; 313971 of Mar. 17, 1885 to T. H. White; 358994 of Mar. 8, 1887 to W. H. Stewart; 359925 of Mar. 22, 1887 to Clinton French; 756180 of Mar. 29, 1904 to J. H. Munro; 1728795 of Sep. 17, 1929 to J. H. Griffin; 1930427 of Oct. 10, 1933 to J. H. Griffin; Can. Pats. 42242 of Mar. 10, 1893 to R. A. Chesebrough; 370937 of Dec. 28, 1937 to Lancy Snow.

p. 800 (776) Ger. Pat. 654211 of Mar. 22, 1934 to D. Anderson & Son, Ltd.

p. 800 (777) U. S. Pat. 2211371 of Aug. 13, 1940 to H. A. Faber.

p. 800 (778) U. S. Pats. 137566 of Apr. 8, 1873 to E. R. Percy; 438973 of Oct. 21, 1890 to J. C. Zallé; Can. Pat. 337819 of Dec. 12, 1933 to Frazzi, Ltd.; Brit. Pat. 389668 of Apr. 28, 1932 to Laurent Noesen; Ger. Pats. 212527 of Oct. 30, 1906 to Ludwig Esselborn; 580391 of Jun. 17, 1930 to Karl Meier; 594470 of Jan. 17, 1928 to Richard Franz and Karl Meier.

p. 800 (779) "Federal Spec. for Surfacing-Material; (for) Bituminous Built-up Roofing," SS-S-791, Federal Standard Stock Catalog, Section IV (Part 5).

p. 801 (780) Ger. Pat. Design 1407419 of May 20, 1937 to Fritz Werner.

p. 801 (781) U. S. Pat. 1977669 of Oct. 23, 1934 to T. M. Dantz; Ger. Pat. 5430 of Sep. 28, 1878 to H. Frühling.

p. 801 (782) Ger. Pat. Design 78074 of Jun. 8, 1897 to Louis Lindenberg.

p. 801 (783) U. S. Pats. 1427755 of Aug. 29, 1922 to W. A. Harris; 2061066 of Nov. 17, 1936 to C. R. Eckert; Can. Pats. 346328 of Nov. 27, 1934 to Barrett Co.; 362228 of Dec. 1, 1936 to Barrett Co.

p. 801 (784) U. S. Pat. 1340347 of May 18, 1920 to C. E. Rahr.

p. 801 (785) U. S. Pats. 1493447 of May 6, 1924 to O. A. Heppes; 1788121 of Jan. 6, 1931 to F. C. Overbury; 2096784 of Oct. 26, 1927 to W. W. Candler; 2122747 of Jul. 5, 1938 to Lester Kirschbraun; Can. Pats. 249096 of Apr. 28, 1925 to Flintkote Co.; 379786 of Feb. 28, 1939 to Patent & Licensing Corp.; Ger. Pat. Design 326523 of Nov. 30, 1907 to Wilhelm Zerlin.

p. 801 (786) U. S. Pat. 1441861 of Jan. 9, 1923 to T. B. Lehon.

p. 801 (787) U. S. Pats. 2250548 of Jul. 29, 1941 to W. H. C. Ness; 2252834 of Aug. 29, 1941 to Hal Callaway; 225156 of Sep. 9, 1941 to C. C. Figge; 2255279 of Sep. 9, 1941 to Hal Callaway; 2271143 of Jan. 27, 1942 to H. D. Martinus; 2274647 of Mar. 3, 1942 to R. T. Avard and F. W. Herring.

p. 801 (788) Ger. Pats. 85901 of Jun. 5, 1895 to Büsscher & Hoffmann G.m.b.H.; Design 60628 of Jul. 13, 1896 to Arthur Siebel; Design 88411 of Jan. 11, 1898 to Ph. Fahnenreißer.

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- p. 811 (851) U. S. Pat. 1774204 of Aug. 26, 1930 to H. L. Levin.
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p. 821 (920) "Federal Spec. for Paper; Kraft Wrapping," UU-P-268b, Federal Standard Stock Catalog, Section IV (Part 5), Feb. 1, 1944.

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p. 822 (927) U. S. Pats. 328957 and 328958 of Oct. 27, 1885 to C. A. Maxfield.

p. 822 (928) U. S. Pats. 347200 of Aug. 10, 1886 to F. C. Robinson and W. H. Cothren; 369700 of Sep. 13, 1887 to W. H. Fay; 1401524 of Dec. 27, 1921 to Joaquin Crespo; 1405220 of Jan. 31, 1922 to J. R. Hyer; 1882212 of Oct. 11, 1932 to J. A. De Cew.

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p. 835 (1032) *Can. Pat.* 322458 of May 17, 1932 to Brown Co.

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- p. 835 (1034) U. S. Pat. 284794 of Sep. 11, 1883 to S. M. Allen; "Bituminized-Fiber Drain and Sewer Pipe," Commercial Standard CS-116-44, National Bureau of Standards, Wash., D. C. (Mar. 10, 1944).
- p. 836 (1035) U. S. Pats. 1277108 of Aug. 27, 1918 to W. D. Pardoe; 1301612 of Apr. 22, 1919 to Sumner Simpson; 1354996 of Oct. 5, 1920 to W. D. Pardoe; 1450319 of Apr. 3, 1923 to Lester Kirschbraun; 1578928 of Mar. 30, 1926 to Sumner Simpson; 1810714 of Jun. 16, 1931 to Lester Kirschbraun; 2138876 of Dec. 6, 1938 to I. J. Novak; Can. Pats. 260958 of May 18, 1928 to Raybestos Co.; 302357 of Jul. 22, 1930 to Raybestos Co.; 321631 of Apr. 19, 1932 to Canadian Raybestos Co., Ltd.
- p. 836 (1036) Can. Pat. 233651 of Aug. 21, 1923 to W. R. Seigle.
- p. 836 (1037) U. S. Pats. 1436362 of Nov. 21, 1922 to W. R. Seigle; 2037189 of Apr. 14, 1936 to J. W. Abernethy, S. J. Smyer and A. B. Kuhn.
- p. 836 (1038) U. S. Pats. 1498386 of Jun. 17, 1924 to Lester Kirschbraun; 2052779 of Sep. 1, 1936 to H. J. Lidkea.
- p. 836 (1039) U. S. Pat. 1275043 of Aug. 6, 1918 to Lester Kirschbraun.
- p. 836 (1040) U. S. Pats. 1270559 of Jun. 25, 1918 to Sumner Simpson; 1712002 of May 7, 1929 to J. A. Heany; 1992601 of Feb. 26, 1935 to W. A. Blume; Reissues 20907 and 20908 of Nov. 1, 1938 to W. A. Blume.
- p. 836 (1041) U. S. Pat. 1941280 of Dec. 26, 1933 to Mark Shoeld.
- p. 836 (1042) U. S. Pat. 2099132 of Nov. 16, 1937 to S. P. Miller.
- p. 836 (1043) U. S. Pat. 1249019 of Dec. 4, 1917 to H. St. L. Buchner.
- p. 836 (1044) U. S. Pats. 2006232 of Jun. 25, 1935 to C. A. Upson; 2043987 of Jun. 16, 1936 to G. B. Brown; 2045384 of Jun. 23, 1936 to William Gerb; 2063964 and 2064327 of Dec. 15, 1936 to C. A. Upson; 2090459 of Aug. 17, 1937 to C. R. Paton; 2110492 of Mar. 8, 1938 to C. A. Upson; 2113128 of Apr. 5, 1938 to G. R. Cunningham; 2116771 of May 10, 1938 to D. R. Seaman; 2124463 of Jul. 19, 1938 to G. R. Cunningham; Design 112568 of Dec. 13, 1938 to Max Skolnik; 2124263 of Jan. 3, 1939 to C. A. Upson; 2173797 of Sep. 19, 1939 to E. A. Toohey and G. J. Campbell; 2180305 of Nov. 14, 1939 to E. O. Groskopf; 2240326 of Apr. 29, 1941 to E. A. Burns; 2320737 of Jun. 1, 1943 to H. B. Hutten; Can. Pats. 366008 of May 11, 1937 to Seaman Paper Co.; 372765 of Mar. 29, 1938 to Burlington Mills, Inc.; 382443 of Jul. 4, 1939 to Burlington Mills, Inc.; 392213 of Oct. 29, 1940 to Lite-O-Tex Products Corp.; 393989 of Jan. 14, 1941 to Woodall Industries, Inc.; Brit. Pats. 449979 of Jun. 26, 1935 to F. B. Dehn; 450183 of Feb. 11, 1935 to William Gerb; French Pat. 828039 of Oct. 19, 1937 to Woodall Industries, Inc.
- p. 836 (1045) U. S. Pats. 1305404 of Jun. 3, 1919 to R. P. Perry; Reissue 15461 of Sep. 26, 1922 to R. P. Perry; 175918 of Oct. 9, 1934 to R. E. Berg; 2008654 and 2008655 of Jul. 16, 1935 to G. W. Clarvoe; 2028950 of Jan. 28, 1936 to M. S. Randall; 2028962 of Jan. 28, 1936 to H. J. Woodall and M. S. Randall; 2106792 of Feb. 1, 1938 to D. S. Bruce and R. E. Berg; 2274792 of Mar. 3, 1942 to A. L. Jennings, W. F. Mitchell and E. A. Worm, Jr.; 2298326 of Oct. 13, 1942 to E. A. Worm, Jr.; Can. Pats. 361149 of Oct. 13, 1936 to Johns-Manville Corp.; 367206 of Jul. 6, 1937 to Johns-Manville Corp.; 414798 of Aug. 31, 1943 to W. F. Hayes.
- p. 836 (1046) Can. Pat. 392514 of Nov. 12, 1940 to Woodall Industries, Inc.
- p. 836 (1047) U. S. Pat. 2192516 of Mar. 5, 1940 to G. R. Cunningham; Can. Pat. 392515 of Nov. 12, 1940 to Woodall Industries, Inc.
- p. 836 (1048) U. S. Pat. 2325594 of Aug. 3, 1943 to H. B. Denman.
- p. 836 (1049) U. S. Pat. 2019233 of Oct. 29, 1935 to G. A. Nicol, Jr.; Can. Pat. 359961 of Aug. 18, 1936 to G. A. Nicol, Jr.
- p. 836 (1050) U. S. Pats. 1541587 of Jun. 9, 1925 to Joseph Regenstein; 1569107 of Jan. 12, 1926 to J. F. White; 1656647 of Jan. 17, 1928 to Joseph Regenstein; 1694523 of Dec. 11, 1928 to J. F. White; 2073894 of Mar. 16, 1937 to F. P. Wood; 2100812 of Nov. 30, 1937 to William Marshall; 2100891 of Nov. 30, 1937 to P. R. Zinser; 2197822 of Apr. 23, 1940 to F. P. Wood; 2303195 of Nov. 24, 1942 to K. E. Buff; Can. Pats. 379169 of Jan. 24, 1939 to F. P. Wood; 399036 of Sep. 2, 1941 to National Automotive Fibres, Inc.; 402217 of Jan. 13,

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1942 to National Automotive Fibres, Inc.; 414799 of Aug. 31, 1943 to W. F. Hayes; Brit. Pat. 476327 of Mar. 2, 1936 to A. R. Wylie.

p. 837 (1051) U. S. Pats. 2147058 of Feb. 14, 1939 to M. S. Randall and G. E. Kasch; 2147059 of Feb. 14, 1939 to M. S. Randall.

p. 837 (1052) U. S. Pats. 2113128 of Apr. 5, 1938 to G. R. Cunningham; 2139882 of Dec. 13, 1938 to G. R. Cunningham; 2184139 and 2184140 of Dec. 19, 1939 to G. R. Cunningham; 2192517 of Mar. 5, 1940 to G. R. Cunningham; Can. Pat. 386224 of Jan. 9, 1940 to Woodall Industries, Inc.; Brit. Pat. 486642 of Jun. 8, 1938 to Woodall Industries, Inc.

p. 837 (1053) Can. Pat. 402216 of Jan. 13, 1942 to National Automotive Fibres, Inc.

p. 837 (1054) U. S. Pats. 1494346 of May 20, 1924 to F. H. Eastman and G. R. Stark; 2063611 of Dec. 8, 1936 to W. J. MacLeod; 2105397 of Jan. 11, 1938 to G. G. Barr and A. H. Kirchner; Can. Pats. 247139 of Feb. 24, 1925 to F. H. Eastman; 248327 of Mar. 31, 1925 to F. H. Eastman and G. R. Stark.

p. 837 (1055) U. S. Pats. 2037205 of Apr. 14, 1936 to L. F. Barnum; 2099910 of Nov. 23, 1937 to Otto Thiel; 2131704 of Sep. 27, 1938 to L. G. Daly and F. X. Meiners; Can. Pat. 375850 of Aug. 16, 1938 to L. F. Barnum.

p. 838 (1056) Ger. Pat. 727252 of Sep. 24, 1942 to J. & O. Krebber A.-G.

p. 838 (1057) Brit. Pat. 412393 of Jun. 28, 1934 to R. E. Horley.

p. 838 (1058) U. S. Pat. 2319933 of May 25, 1943 to E. G. Kerr.

p. 838 (1059) "Standard Specs. for Friction Tape for General Use for Electrical Purposes" (D 69-38), A.S.T.M. Standards 1942, III, 485; EA-D 694, Jul. 20, 1943; "Standard Specs. for Rubber Insulating Tape" (D 119-38), A.S.T.M. Standards 1942, III, 490, EA-D 119, Nov. 25, 1942; Approved as A.S.A.No.: C 59.6-1939 by the American Standards Association; "Tentative Methods of Testing and Tolerances for Woven Tapes" (D 259-42T), A.S.T.M. Standards 1942, III, 1498; "Standard Specifications and Methods of Test for Asbestos Tape for Electrical Purposes" (D 315-41), A.S.T.M. Standards 1942, III, 576; "Standard Specifications for 0.007-in. Cotton Tape for Electrical Purposes" (D 335-36), A.S.T.M. Standards, III, 593; "Federal Spec. for Tape; Friction," HH-T-101a, Federal Standard Stock Catalog Section IV (Part 5), Dec. 28, 1939; E-HH-T-101a, May 14, 1943; "Adhesive Insulating Tape for Electrical Purposes," War Emergency British Standard No.: 1078-1942, British Standards Institution, London.

p. 839 (1060) U. S. Pat. 1842616 of Jan. 26, 1932 to I. W. Levine.

p. 839 (1061) Can. Pat. 340705 of Apr. 10, 1934 to W. W. McLaurin.

p. 839 (1062) Ger. Pat. 682667 of Sep. 28, 1939 to Vereinigte Korkindustrie, A.-G.

p. 839 (1063) Dutch Pat. 51356 of Nov. 15, 1941 to Cornelis Vos and Hubertus G. M. van Leeuwen.

p. 839 (1064) "Study of Commercial Wall Boards," by F. C. Clark and A. D. Conley, *Paper*, 25, No. 23 (Feb. 11, 1920); "Properties of Fiber Building Boards," by C. G. Weber, F. T. Carson and L. W. Snyder, Misc. Publication No. 132, Bureau of Standards, Wash., D. C. (1931); "Federal Spec. for Wall-Board; Composition," UU-W-101a, Federal Standard Stock Catalog, Section IV (Part 5), Jul. 3, 1935.

p. 839 (1065) "Binding Agents in the Manufacture of Fiberboards," by A. Wilke, *Holz Roh- u. Werkstoff*, 5, 78 (1942); U. S. Pats. 1033756 of Jul. 23, 1912 to W. G. Fiske; 1292067 of Jan. 21, 1919 to H. H. Robertson; 1353323 of Sep. 21, 1920 to F. B. Davidson; 1503957 of Aug. 5, 1924 to Otto Kress; 1689812 of Oct. 30, 1928 to F. P. Wood; 1694523 of Dec. 11, 1928 to J. F. White; 1905397 of Apr. 25, 1933 to C. G. Reynolds; 1910671 of May 23, 1933 to S. J. Blum; Brit. Pats. 540326 of Apr. 8, 1940 to Victor Lefebure, A. H. Douglas, J. J. Etridge and Imperial Chemical Industries, Ltd.; 551380 of Aug. 19, 1941 to S. Bunton, W. Crawford and J. K. Paterson.

p. 840 (1066) U. S. Pats. 269786 of Dec. 26, 1882 to S. H. Hamilton; 1033756 of Jul. 23, 1912 to W. G. Fiske; 1541587 of Jun. 9, 1925 to Joseph Regenstein; 1569107 of Jan. 12, 1926 to J. F. White; 1656647 of Jan. 17, 1928 to Joseph Regenstein; 1694523 of Dec. 11, 1928 to J. F. White; 1999385 of Apr. 30, 1935 to H. J. Woodall; 2073894 of Mar. 16, 1937 to

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F. P. Wood; Reissue 20943 of Dec. 6, 1938 to H. J. Woodall; *Brit. Pat.* 476327 of Mar. 2, 1936 to A. R. Wylie (corresponds to U. S. Pat. 2073894 of Mar. 16, 1937 to F. P. Wood).

p. 840 (1067) U. S. Pats. 269786 of Dec. 26, 1882 to S. H. Hamilton; 631742 of Aug. 22, 1899 to O. L. Gardner; 1248181 of Nov. 27, 1917 to B. W. Sidwell; 1477532 of Dec. 18, 1923 to C. S. Bird; 2075317 of Mar. 30, 1937 to C. A. Upson.

p. 840 (1068) U. S. Pat. 1479044 of Jan. 1, 1924 to Lester Kirschbraun.

p. 840 (1069) U. S. Pat. 2215245 of Sep. 17, 1940 to G. D. King and J. W. Gill; *Can. Pat.* 398041 of Jul. 22, 1941 to G. D. King and J. W. Gill.

p. 840 (1070) U. S. Pat. 2215246 of Sep. 17, 1940 to J. W. Gill.

p. 840 (1071) U. S. Pat. 2300137 of Oct. 27, 1942 to A. C. Salisbury.

p. 840 (1072) U. S. Pats. 195483 of Sep. 25, 1877 to J. M. Cobb; 1074829 of Oct. 7, 1913 to C. S. Bird and G. R. Wyman.

p. 840 (1073) U. S. Pats. 1336403 and 1336404 of Apr. 6, 1920 to H. F. Weiss; 1353619 of Sep. 21, 1920 to H. W. White; 1571667 of Feb. 2, 1926 to O. A. Heppes; 1578663 of Mar. 30, 1926 to H. C. Innes; 2278146 of Mar. 31, 1942 to W. J. Moeller.

p. 840 (1074) U. S. Pat. 2316467 of Apr. 13, 1943 to O. C. H. Sturken.

p. 840 (1075) *Brit. Pat.* 554562 of Nov. 26, 1941 to J. Bennie.

p. 840 (1076) *Brit. Pat.* 540326 of Oct. 14, 1941 to Victor Lefebure, A. H. Douglas, J. J. Etridge and Imperial Chemical Industries, Ltd.

p. 840 (1077) U. S. Pats. 1115593 of Nov. 3, 1914 to J. P. Sexton; Reissue 14148 of Jun. 5, 1916 to J. P. Sexton; 1205360 and 1205399 of Nov. 21, 1916 to J. P. Sexton; 1325883 of Dec. 23, 1919 to J. P. Sexton; *Brit. Pat.* 558711 of May 13, 1942 to Imperial Chemical Industries, Ltd.

p. 840 (1078) U. S. Pat. 1793810 of Feb. 24, 1931 to H. L. Levin; *Can. Pat.* 320383 of Mar. 8, 1932 to Flintkote Co.

p. 840 (1079) U. S. Pats. 1831058 of Nov. 10, 1931 to H. A. Cumfer; 2075373 of Mar. 30, 1937 to John Tomec; Reissue 20607 of Dec. 28, 1937 to John Tomec.

p. 840 (1080) U. S. Pat. 1821120 of Sep. 1, 1931 to H. M. Spencer.

p. 840 (1081) U. S. Pat. 1874674 of Aug. 30, 1932 to C. A. Watson.

p. 840 (1082) U. S. Pats. 1322278 of Nov. 18, 1919 to M. K. Armstrong; 1417836 of May 30, 1922 to Lester Kirschbraun; 1644652 of Oct. 4, 1927 to Lester Kirschbraun.

p. 840 (1083) U. S. Pats. 985140 of Feb. 28, 1911 to Hedley Button; 1292067 of Jan. 21, 1919 to H. H. Robertson; 1322278 of Nov. 18, 1919 to M. K. Armstrong; 1362888 of Dec. 21, 1920 to T. J. Mullin; 1902298 of Mar. 21, 1933 to H. C. Avery and Lester Kirschbraun; *Can. Pat.* 278743 of Mar. 20, 1928 to Flintkote Co.; *Brit. Pats.* 121205 of Dec. 21, 1917 to D. L. Irwin and E. R. James; 138428 of Feb. 11, 1919 to George Harrison.

p. 840 (1084) U. S. Pat. 1489567 of Apr. 8, 1924 to H. F. Weiss; *Can. Pat.* 303706 of Sep. 9, 1930 to Building Products, Ltd.

p. 840 (1085) U. S. Pat. 1510233 of Sep. 30, 1924 to O. D. McFarland.

p. 840 (1086) U. S. Pats. 206850 of Aug. 13, 1878 to D. S. Armstrong; 1473981 of Nov. 13, 1923 to J. W. Wagner.

p. 840 (1087) U. S. Pat. 2290833 of Jul. 21, 1942 to P. V. Keyser, Jr. and W. E. Spels. house.

p. 841 (1088) "Federal Spec. for Fiberboard; Insulating," LLL-F-321b, Federal Standard Stock Catalog, Section IV (Part 5), Jun. 30, 1942, Amendment-1, Dec. 17, 1942.

p. 841 (1089) *Brit. Pat.* 477919 of Jan. 10, 1938 to Kaj Branning and Walter Henriksen.

p. 841 (1090) *Ger. Pat.* 68965 of Jul. 12, 1892 to Ernst Biernath.

p. 841 (1091) U. S. Pats. 1352619 of Sep. 21, 1920 to H. W. White; 1461337 of Jul. 10, 1923 to H. F. Weiss; 1680144 of Aug. 7, 1928 to A. C. Fischer; 2012805 of Aug. 27, 1935 to A. G. Brown and S. E. McPartlin; 2123696 of Jul. 12, 1938 to A. C. Fischer; 2124921 of Jul. 26, 1938 to Maximilian Lederer; *Brit. Pat.* 486645 of Oct. 22, 1937 to Theodor Dieden and Nils Ryberg; *Ger. Pat.* 651929 of Mar. 30, 1935 to Norddeutsche Asbest- und Gummiwerke Kurt Weber & Co., G.m.b.H.

p. 841 (1092) U. S. Pats. 688420 of Dec. 10, 1901 to George Kelly; 737099 of Aug. 25,

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1903 to C. C. Hall; 811778 of Feb. 6, 1906 to C. C. Hall; 945583 of Jan. 4, 1910 to T. B. Parkison; 1204149 of Nov. 7, 1916 to O. E. Gelertsen; 1242537 of Oct. 9, 1917 to William Fay; 1336403 and 1336404 of Apr. 6, 1920 to H. F. Weiss; 1394610 of Oct. 25, 1921 to J. A. DeCew; 1656828 of Jan. 17, 1928 to E. R. Powell; 1928264 of Sep. 26, 1933 to E. R. Powell; 1948395 of Feb. 20, 1934 to E. R. Powell; Reissue 19627 of Jun. 25, 1935 to E. R. Powell; 2055446 of Sep. 22, 1936 to E. R. Powell; 2163567 of Jun. 20, 1939 to J. H. Gregory; 2223086 of Nov. 26, 1940 to R. C. Williams and J. F. Hall; 2282230 of May 5, 1942 to W. M. MacAlpine; Reissue 22090 of May 5, 1942 to E. R. Powell; 2305516 of Dec. 15, 1942 to H. T. Coss and W. M. MacAlpine; 2319666 of May 18, 1943 to D. C. Drill; 2343600 and 2343601 of Mar. 7, 1944 to A. F. Weimann; 2338839 of Jan. 11, 1944 to H. T. Coss; *Can. Pat.* 412296 of Aug. 3, 1943 to Johns-Manville Corp.; 420920 and 420921 of Jun. 20, 1944 to Allied Chemical & Dye Corp.; *Brit. Pats.* of 1887 (Sep. 8), 12179 to Charles Jackson; 549973 of Jan. 11, 1941 to Stillite Products, Ltd. and E. E. Gaunt; *Ger. Pat.* 709938 of Jul. 17, 1941 to Bruno Neuhof.

p. 841 (1903) "Insulating Plates from Spun Slags," by B. Telnov, *Novosti Tekhniki*, 10 (No. 11), 34 (1941); *U. S. Pats.* 1949087 of Feb. 27, 1934 to M. D. Squiers; 2162386 of Jun. 13, 1939 to Bruno Neuhof; 2307117 of Jan. 5, 1943 to D. C. Drill; *Can. Pat.* 419525 of Apr. 11, 1944 to Leroy Jennings and H. C. Brown, Jr.; *Brit. Pats.* 470675 of Oct. 1, 1935 to N. V. Maatschappij tot Beheer en Exploitatie van Octrooien; 550833 of Jun. 11, 1941 to Stillite Products, Ltd. and E. E. Gaunt; 556944 of Mar. 23, 1944 to Armstrong Cork Co.; *Ger. Pats.* 650652 of Jul. 5, 1934 to Bruno Neuhof; 711863 of Sep. 11, 1941 to Deutsche Eisenwerke, A.-G.; 714785 of Nov. 13, 1941 to Berliner Gypswerke L. Mundt, vorm. H. Kühne; *French Pat.* 776920 of Feb. 7, 1935 to Diafan S. A.; *Dutch Pat.* 50780 of Aug. 15, 1941 to N. V. Maatschappij tot Beheer en Exploitatie van Octrooien.

p. 841 (1904) "Federal Spec. for Mineral-Wool, Impregnated; Blanket, Block, and Pipe-Covering (Molded), (for Low Temperatures)," HH-M-371, Federal Standard Stock Catalog, Section IV (Part 5), Oct. 29, 1938; *U. S. Pat.* 2338839 of Jun. 11, 1944 to H. T. Coss; *Can. Pat.* 420817 of Jun. 13, 1944 to Johns-Manville Corp.

p. 841 (1905) *U. S. Pats.* 2288072 of Jun. 30, 1942 to H. W. Collins; 2350996 of Jun. 13, 1944 to W. A. Atkinson and H. W. Collins.

p. 841 (1906) *Can. Pat.* 411722 of Apr. 13, 1943 to E. A. I. Orrmell.

p. 841 (1907) *Brit. Pats.* 535749 of Apr. 21, 1941 to British Plaster Board, Ltd. and L. F. Allsop; 548706 of Apr. 18, 1941 to Gyproc Products, Ltd. and J. F. Strable.

p. 841 (1908) *U. S. Pats.* 845290 of Feb. 26, 1909 to E. H. Binns; 891428 of Jun. 23, 1908 to W. H. Latus; 958450 of May 17, 1910 to H. R. Wardell; 999951 of Aug. 8, 1911 to C. S. Bird; 1519280 and 1519281 of Dec. 16, 1924 to Kurt Wandel; 1520284 of Dec. 23, 1924 to G. H. Ellis; 1574886 of Mar. 2, 1926 to O. A. Heppes and W. H. Cady; 1640619 of Aug. 30, 1927 to C. W. Scoggin; 1765796 of Jun. 24, 1930 to Lester Kirschbraun; 1772686 of Aug. 12, 1930 to C. E. Rahr; 1802880 of Apr. 28, 1931 to H. A. Cumfer; 1949255 of Feb. 27, 1934 to C. L. Keller; 2042586 of Jun. 2, 1936 to John Campbell and R. G. Quinn; 2215241 of Sep. 17, 1940 to G. R. Eichelberger and C. D. Alteck; *Can. Pats.* 133756 of Jun. 13, 1911 to Hermann Schlisske; 251757 of Jul. 14, 1925 to Gardner & Lewis; 256565 of Dec. 22, 1925 to Flintkote Co.; 329434 of Jan. 17, 1933 to Bird & Son, Inc.; 355645 of Jan. 28, 1936 to International Paper Co.; *Brit. Pats.* of 1912 (May 6), 10695 to E. H. Angier; 374298 of Jun. 9, 1932 to Otto Goy; 431162 of Jul. 2, 1935 to John Campbell and R. G. Quinn; *Ger. Pats.* Design 333255 of Feb. 12, 1908 to L. Wunnenberg; Design 415347 of Feb. 26, 1910 to August Preuss; Design 446545 of Nov. 5, 1910 to Bremer Papier und Wellpappenfabrik, A.-G.

p. 841 (1909) *Brit. Pats.* 550561 and 550562 of Apr. 8, 1940 to Munters' Ind. Aktieab.; 550646 of Jun. 6, 1940 to Munters' Ind. Aktieab.

p. 841 (1100) *U. S. Pat.* 2206962 of Jul. 9, 1940 to H. C. Karcher; *Can. Pats.* 375862 of Aug. 16, 1938 to M. S. Gazelle; 376675 of Sep. 20, 1938 to H. E. Wilkins.

p. 841 (1101) *Can. Pat.* 412529 of May 18, 1943 to C. G. Munters.

p. 841 (1102) *U. S. Pats.* 403588 of May 21, 1889 to G. A. Herdman; 527505 of Oct. 16, 1894 to Patrick Norton; 540992 of Jun. 11, 1895 to A. E. Krause; 836157 of Nov. 20, 1906 to P. W. Turner; 1015919 of Jan. 30, 1912 to H. R. Wardell; *Brit. Pats.* 338403 of Dec. 13,

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1928 to E. Rudin and Heinrich Kollbrunner; 531059 of Jul. 11, 1939 to Ardor Eng. Co., Ltd. and G. Richardson; *Ger. Pat.* Design 1338133 of Apr. 26, 1935 to Heinrich Züge.

p. 841 (1103) *U. S. Pat.* 1587462 of Jun. 1, 1926 to F. W. Adams; *Can. Pat.* 378577 of Dec. 27, 1938 to Woodall Industries, Inc.

p. 841 (1104) *U. S. Pats.* 1449221 of Mar. 20, 1923 to G. H. Ellis; 1457664 of Jun. 5, 1923 to S. M. Ford; 1900699 of Mar. 7, 1933 to G. H. Ellis; 1926737 of Sep. 12, 1933 to S. J. Blum; 1941769 of Jun. 2, 1934 to G. J. Ward; 2036466 of Aug. 7, 1936 to G. H. Ellis; 2134659 of Oct. 25, 1938 to G. H. Ellis; *Can. Pats.* 233378 of Aug. 7, 1923 to Barrett Co.; 281500 of Jul. 3, 1928 to C. W. Scoggin and Niels Nissen; 389326 of Jun. 11, 1940 to Insulite Co.; *Ger. Pats.* 112629 of May 5, 1899 to A. W. Andernach; 121436 of May 6, 1899 to A. W. Andernach.

p. 841 (1105) *U. S. Pat.* 1532084 of Mar. 31, 1925 to J. K. Shaw; *Brit. Pat.* 515045 of Apr. 21, 1938 to Celotex Corp.

p. 841 (1106) *U. S. Pats.* 2265244 of Dec. 9, 1941 to C. L. Neumeister; 2301215 of Nov. 10, 1942 to H. C. Koch.

p. 841 (1107) *U. S. Pat.* 2274495 of Feb. 24, 1942 to C. G. Muench.

p. 841 (1108) *U. S. Pat.* 2299144 of Oct. 20, 1942 to C. C. Heritage and H. E. Walter.

p. 841 (1109) *U. S. Pats.* 1595673 of Aug. 10, 1926 to G. R. Magney and W. R. Nelson; 1598981 of Sep. 7, 1926 to W. R. Nelson and T. B. Hennessey; Reissue 16753 of Oct. 4, 1927 to W. E. Nelson; 1941769 of Jan. 2, 1934 to G. J. Ward.

p. 842 (1110) *U. S. Pat.* 1938351 of Dec. 5, 1933 to E. S. Penn.

p. 842 (1111) *U. S. Pats.* 1822632 of Sep. 8, 1931 to H. F. Winkelmann; 2111219 of Mar. 15, 1938 to W. N. Mayo; 2289250 of Jul. 7, 1942 to P. S. Denning; 2326889 of Aug. 17, 1943 to K. W. Schulz and C. G. Schulz; 2326896 of Aug. 17, 1943 to R. E. Sprague and H. M. Sprague; *Can. Pat.* 324493 of Jul. 26, 1932 to H. F. Winkelmann.

p. 842 (1112) *U. S. Pat.* 1152798 of Sep. 7, 1915 to Julius de Long and J. B. d'Homergue.

p. 842 (1113) *U. S. Pats.* 1801525 of Apr. 21, 1931 to W. E. Nelson; 1917456 of Jul. 11, 1933 to A. O. Mickelson.

p. 842 (1114) *U. S. Pats.* 1945308 of Jan. 30, 1934 to A. C. Fischer; 2134495 of Oct. 25, 1938 to H. J. Woodall and M. S. Randall.

p. 842 (1115) *U. S. Pat.* 2035122 of Mar. 24, 1936 to S. C. Fulton and Vladimir Kalichevsky.

p. 842 (1116) *U. S. Pats.* 2170655 of Aug. 22, 1939 to C. A. Fourness; 2339326 of Jan. 18, 1944 to C. A. Fourness and J. B. Catlin.

p. 842 (1117) *U. S. Pat.* 2291140 of Jul. 28, 1942 to C. W. Bowyer.

p. 842 (1118) *U. S. Pats.* 2148167 of Feb. 21, 1939 to H. T. Lyman; 2264546 of Dec. 2, 1941 to S. A. Ochs.

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45836; p. 765 (449)	81712; p. 790 (666)	101732; p. 791 (679)
48172; p. 765 (447), p. 770 (494)	81768; p. 787 (643)	101921; p. 793 (688)(689)
49171; p. 763 (436), p. 771 (514)	83704; p. 790 (673)	102555; p. 798 (749)
51438; p. 765 (452)	83718; p. 791 (677)	104095; p. 787 (641)
52538; p. 765 (455)	84668; p. 790 (666)	104948; p. 791 (681)
53015; p. 779 (566), p. 790 (658)(674)	84808; p. 793 (688)	104971; p. 790 (665)
53086; p. 781 (587)	85638; p. 790 (666)	105124; p. 777 (560)
53087; p. 781 (587)	85639; p. 790 (666)	105276; p. 790 (671)
54160; p. 790 (665)	86085; p. 790 (666)	107209; p. 787 (644)
54917; p. 785 (632)	86194; p. 791 (680)	107312; p. 790 (671)
56106; p. 777 (560)	87057; p. 790 (666)	109077; p. 774 (536)
57126; p. 791 (682)	87104; p. 787 (641)	112568; p. 836 (1044)
61363; p. 790 (671)	88995; p. 787 (643)	120622; p. 774 (540)
67565; p. 790; p. 790 (666)	89471; p. 791 (681)	131018; p. 793 (689)
68522; p. 787 (639)	89639; p. 790 (666)	131154; p. 793 (689)
68552; p. 791 (676)	89783; p. 790 (658)(666)	133920; p. 793 (689)
70986; p. 791 (679)	90115; p. 782 (603)	133925; p. 793 (689)
70987; p. 791 (679)	91061; p. 791 (680)	135035; p. 791 (681)
	91654; p. 787 (652)	135045; p. 791 (681)
	91744; p. 779 (576)	135475; p. 793 (689)
	92132; p. 791 (680)	135476; p. 793 (689)
	92379; p. 782 (603)	135477; p. 793 (689)

